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AN ACCOUNT
OF THE
LATEST DISCOVERIES IN CHEMISTRY,
WITH
EXTRACTS
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WITH
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OF
D I S S E R T A T I O N S

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Jos. Banks

I. ON THE
ACID OF FRUITS AND BERRIES;

By CH. WILLIAM SCHEELE.

§. I.

SINCE I made my experiments on the nature and properties of the pure acid of lemons, I have examined the juices of various other fruits and berries, and especially of those which are indigenous with us, in order to see whether, and how far their acids agree in their properties with the acid of lemons. In these experiments, I generally employed the juices of the unripe berries; and, to obtain the acid in greater abundance, I mashed the fruits in a wooden mortar, and afterwards filtered the expressed juice through blotting paper.

§. II.

Neither the juice of gooseberries (*Ribes grossularia*) nor the juices of any other berries and fruits which I have examined, con-

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tain

tain any acid of sorrel, as may be inferred from this circumstance, viz. that they do not precipitate lime-water*.

(a) I saturated some gooseberry juice with chalk, filtrated it into a matrafs, and set it upon the fire. After it had boiled for a minute, there was a copious precipitation of a white sediment. (b) The clear liquor, which remained over this sediment, I poured through a filter into another matrafs, and boiled it again for a few minutes; but no more precipitation took place: in this respect, therefore, the juice in question exactly resembles the juice of lemons. (c) Now, in order to obtain this acid likewise in a crystallized form, I poured some diluted vitriolic acid upon the lixiviated sediment, or limoniated calcareous earth, *calx citrata*, and in other respects, proceeded in the manner described in my paper on the acid of lemons. Thus I obtained the acid of goose-

* Though some acid juices may precipitate lime-water, not in consequence of containing acid of sorrel, but acid of apples (§. 5.) yet such a precipitate is re-dissoluble on the addition of vinegar; which, however, is not the case with the acid of sorrel.

berries in the form of crystals, which, in every circumstance accorded with the crystallized lemon-juice.

§. III.

(a) The clear liquor, §. 2. b. (though in consequence of this treatment it had now acquired a brown colour) which, during the boiling, had deposited limoniated calcareous earth, was discovered, on applying paper stained with litmus, to contain superfluous acid, of which I had not been able to deprive it by the addition of more chalk; and it, moreover, held a considerable portion of chalk in solution; an effect which could not proceed from the acid of lemons, since limoniated calcareous earth is almost totally insoluble in boiling water: Besides the acid of lemons, therefore, the juice of gooseberries must also contain some other acid. Accordingly, I endeavoured to bring forth this concealed acid in its pure state, that I might see how far it agreed with other known acids.

After some attempts, I found, that on mixing highly rectified spirit of wine with this liquor, a coagulum was formed, which

contained this acid united with calcareous earth; but as some gummy matter, equally insoluble in spirit of wine, was also contained in this coagulum, it was necessary that I should separate the gummy part from the juice previously to its saturation with chalk. (b) I therefore evaporated some gooseberry-juice to the consistence of honey, dissolved it in strong spirit of wine, and strained the whole through a paper filter; thus the spirit, together with the acid which was dissolved in it, passed through the paper, whilst the gummy matter was left behind upon the filter. I evaporated this spirit of wine, and to the acid which now remained behind, I added twice as much water. (c) I then saturated it with chalk, and boiled the solution a minute or two, that the limoniated calcareous earth might separate, and afterwards filtered off the clear liquor. Here I had the same calcareous solution as before (a), except that it was freed from the gummy or mucilaginous matter. To this solution I added a sufficient quantity of strong spirit of wine, whereupon a thickening or coagulation took place;

place; after which, I passed the whole through a strainer. The spirit which now dropped through the strainer, held nothing in solution but a little saponaceous and saccharine matter. Upon the coagulum which remained on the filter, I poured some fresh spirit of wine, in order to lixivate it. This coagulum is the substance which I am now to examine.

§. IV.

(a) If a little of it, while it is yet moist, is spread upon the nail, it soon liquefies, but immediately afterwards dries again, with a gloss which equals the finest varnish. (b) It dissolves very readily in water, and reddens paper stained with litmus (§. 3. a.) After this solution has stood a few days exposed to the air, it deposits a number of small crystals, which require boiling water for their solution; and this is found to be a perfectly neutralized solution, from which the calcareous earth is precipitated on adding an alkali. (c) By exposure to the fire in a crucible, this neutral salt is quickly decomposed, the residuum being found to be aerated lime. (d) Now, in or-

der to separate the acid from the calcareous earth, diluted vitriolic acid was added to the solution (b), till no more gypsum or senile was precipitated; and the acid was then filtered off. (e) But finding this method to be attended with some difficulty, by reason that the acid would not completely let go the calcareous earth, and perceiving, moreover, that the acid had a stronger affinity to the calx of lead, than to calcareous earth; I had recourse, in this case, to the same means which I employed for the separation of the acid from the salt of sorrel, viz. vinegar of lead, and vitriolic acid*. Thus I obtained this species of acid, in a pure and perfect state. But, before I describe the properties of this acid, I shall relate the experiments which I have made upon several other juices of fruits, and their acids.

§. V.

The acid of lemons is not present in the juices of any sort of sour apples, whether ripe or unripe; for, after being saturated with chalk and boiled, they yield no sedi-

* See the 2d No. of this Journal, p. 108 & 109.

ment;

ment: but if spirit of wine is added to these juices, after they have been saturated with chalk and filtered, a very considerable coagulation or precipitation ensues. As there is no perceivable gummy matter in the apple-juice, it does not require any preparatory treatment; accordingly, the coagulum which is formed by the addition of spirit of wine, consists entirely of calcareous earth, combined with acid of apples; and this neutral salt shews in all respects exactly the same properties as the salt, §. 4. a. c.

Now, as I could get the acid of gooseberries in greater abundance from the apple-juice, I fell upon a shorter method of proceeding; viz. I saturated the juice with salt of tartar, added vinegar of lead till no more precipitation ensued, and upon the edulcorated precipitate I poured as much diluted vitriolic acid as was requisite, till the mixture had a perfectly acid taste, without any sweetness*; and lastly, I separated the acid,

* As long as a sufficient quantity of vitriolic acid is not added, there still remains a little of the calx of lead dissolved in an excess of acid of apples; and hence proceeds the sweet taste.

by passing it through a strainer, from the vitriol of lead.

Notwithstanding that this acid is contained in the juices of almost all our four fruits, but frequently mixed with acid of lemons (which last acid, however, is not found in the juice of apples) I shall henceforth call it the *Acid of Apples*.

§. VI.

I have found that the juices of the following berries contain a great quantity of acid of lemons, but little or no acid of apples:

Cranberries. *Vaccinium oxycoccos*.

Red Whortleberries. *Vaccinium vitis idæa*.

Birdcherries. *Prunus padus*.

Bittersweetberries. *Solanum dulcamara*.

Hips. *Cynosbatus* (*Rosa canina*)

On the other hand, the following show few or no signs of the acid of lemons, but consist of the acid of apples:

Barberries. *Berberis vulgaris*.

Elderberries. *Sambucus nigra*.

Sloes. *Prunus spinosa*.

Serviceberries. *Sorbus aucuparia*.

Plumbs. *Prunus domestica*.

Lastly,

Lastly, the following berries contain pretty nearly equal quantities of both acids:

Gooseberries. *Ribes grossularia*.

Currants, white, red, and black. *Ribes rubrum*.

Bilberries. *Vaccinium myrtillus*.

Beamberries. *Crataegus aria*.

Cherries. *Prunus cerasus*.

Strawberries. *Fragaria vesca*.

Cloudberry. *Rubus chamaemorus*.

Raspberries. *Rubus idæus*.

But the acid of unripe grapes and of tamarinds consists entirely of the acid of lemons. Lemon-juice, too, shows some traces of acid of apples.

§. VII.

The acid of apples shows the following properties:

(a) It is not susceptible of crystallization, but constantly remains in a fluid state.

(b) With all the three alkalis it affords deliquescent neutral salts.

(c) When perfectly saturated with calcareous earth, it yields small irregular crystals, which require a large quantity of boiling water

water for their solution; but if the acid is a little predominant, the crystals are then readily soluble in cold water.

(d) With the heavy or barytic earth, the effects are the same as with the calcareous earth.

(e) With the aluminous earth it affords a neutral salt which is very difficultly soluble in water.

(f) The earth of magnesia forms with it a deliquescent neutral salt.

(g) Iron gives a brown solution, which is not crystallizable.

(h) Zinc yields beautiful crystals with this acid; but upon the other metals it has not any perceptible effect.

Although the acid of lemons agrees with the acid of apples in many respects, yet in the following circumstances there is a striking difference between them.

(1) Pure acid of lemons shoots into fine crystals; but the acid of apples is not crystallizable.

(2) By means of nitrous acid, the acid of apples may very easily be converted into acid
of

of sugar, or acid of sorrel; but notwithstanding all the pains I have taken, I have never been able to effect this change upon the acid of lemons, even though I employed for its dephlogistification smoking nitrous acid.

(3) Limoniated calcareous earth is almost insoluble in boiling water; whereas the compound of calcareous earth and acid of apples is by far more soluble.

(4) The acid of apples precipitates quicksilver, lead and silver from their solutions in nitrous acid, and likewise gold from its solution in aqua regia diluted with distilled water—in a revived state; but the acid of lemons produces no sort of change in these metallic solutions.

(5) The acid of apples has a weaker attraction to calcareous earth than the acid of lemons; for on boiling limoniated volatile alkali for a minute in a solution of chalk in acid of apples, limoniated calcareous earth is immediately precipitated.

§ VIII.

How frequently have chemists prepared the acid of sugar, without ever attending to the acid which is obtained before the saccharine acid makes its appearance? If nitrous acid be distilled with an equal quantity of sugar, till the mixture begins to incline to a brown colour (which is a sign that all the nitrous acid has been abstracted from it) this syrup will be found to have a considerably acid taste; and after all the saccharine acid is separated from it by means of lime-water, there nevertheless still remains another acid, which neither shows any marks of nitrous acid, nor precipitates lime-water. If this acid be saturated with chalk, the solution filtrated, and strong spirit of wine be mixed with it, a thickening or coagulation takes place. If the vinous spirit be again separated from the curdled matter by filtration, and the coagulum, previouslyedulcorated with fresh spirit of wine, be dissolved in distilled water, and vinegar of lead be added to it, the calx of lead will be precipitated. Lastly,

ly, if the acid is expelled from the calx of lead by means of diluted vitriolic acid, we obtain an acid, which, in its properties, differs not in the least degree from the acid of apples, and consequently is the very same acid. In preparing the acid of sugar, therefore, we produce two acids which exist ready formed in the vegetable kingdom, viz. the acid of apples, and the acid of forrel.

After evaporating the vinous spirit which dropped through the filter from the above-mentioned coagulum, I had a residuum which consisted of a bitter substance, that had lost all its sweetness, and was very deliquescent. It exactly resembled the saponaceous matter in the acid of lemons. I once distilled it with a little nitrous acid, whereby I obtained again acid of apples, and acid of sugar.

§ IX.

Having entered upon this field of enquiry, I resolved to see what results various other substances, as well animal as vegetable, would give, when treated, in like manner, with nitrous acid,

(1) *Gum*

(1) *Gum arabic* yielded both acid of apples, and acid of sugar.

(2) *Manna* did the same.

(3) *Sugar of milk* yielded three kinds of acid, in one and the same process; viz. acid of apples, acid of sugar, and acid of sugar of milk.

(4) *Gum tragacanth*, during its digestion with nitrous acid, let fall a white powder, which was acid of sugar of milk; it afterwards yielded acid of apples and acid of sugar, together with a little calcareous earth, combined with acid of apples.

(5) *Starch* leaves in the nitrous acid, an undissolved substance, which, after it has been separated from the solution by filtration, and has been thoroughlyedulcorated with water, has the appearance of a thick oil, which resembles tallow, but which dissolves very readily in spirit of wine. On distilling this oil by itself, there pass over into the receiver an acid similar to vinegar, and an oil which smells like tallow, and which thickens or congeals in the cold. As to the rest, starch affords both acid of sugar, and acid of apples.

(6) *Potatoe-*

(6) *Potatoe-meal* yields the same results as sugar, together with a little *saccharated** calcareous earth.

(7) *Salep* afforded more *saccharated* calcareous earth, together with acid of apples and acid of sugar.

(8) The *watery extract of aloes* yields, in like manner, acid of sugar, and acid of apples; and loses, at the same time, most of its bitter taste. A good deal of resinous matter, of a deep red colour, separates from the rest, and, during the boiling with the nitrous acid, emits an agreeable odour, very much like that of the flowers of benzoin. Hence, on distilling this resin, I expected to get a sublimate of the same nature as the flowers of benzoin; but scarcely had the retort become hot, when the resin took fire, so that nothing was afterwards found in the retort but a coally matter.

(9) *Extract of Colocynth* was converted by the nitrous acid into a resin, and gave very few marks of saccharine acid.

* By *saccharated* calcareous earth is meant calcareous earth combined with the acid of sugar. This term is employed to prevent circumlocution, and to prevent also, in some measure, the too frequent recurrence of the word acid. TRANSLATOR.

(10) *Extract*

(10) *Extract of Peruvian bark* and several other extracts universally afforded acid of apples, and acid of sugar. They also yield a little salt of sorrel, which proceeds from the neutral salts always contained in these extracts.

(11) An infusion of roasted *coffee*, evaporated to the consistence of a syrup, yields both sorts of acids.

(12) An *infusion of rhubarb*, treated in the same manner, affords a good deal of resinous matter, of a pleasant smell, together with acid of sugar, and acid of apples.

(13) From *opium* a good deal of resin is likewise separated, and it yields, in like manner, both acids.

(14) *Infusion of galls*, too, yields both acids.

(15) All the *distilled or essential oils* which I have treated with nitrous acid, yield very little or no acid of apples, or acid of sugar; except the *oil of parsley seed*, which is completely resolvable into both those acids.

§. X.

From animal substances I was not able to extract these two vegetable acids, as long as
I em-

I employed (which I did at first) diluted nitrous acid; but when I afterwards made use of concentrated nitrous acid, I immediately succeeded in decomposing those substances.

Upon one part of *glue* were poured two parts of smoking nitrous acid, and the mixture was placed in a sand-bath. The glue was quickly dissolved with vehemence, and with an extrication of deep red vapours. The next day, when the solution was cold, there appeared in it a quantity of crystals, which I found to be acid of sugar. The remaining acid, which would not crystallize, I saturated with chalk, from which I afterwards obtained, in the manner before described (§. 5) acid of apples.

Isinglass, white of egg, yolk of egg, and blood, yielded the same results. From all these substances a thick oil or fat is separated, and especially from the yolk of egg; and it is further remarkable, that, if the air which is generated during the process, is collected and examined, it is found to consist of a little fixed air, much foul or phlogisticated air,

and very little nitrous air. Such phlogisticated air, I have never observed to be extricated in the preparation of saccharine acid from the vegetable kingdom; nor do I know that it has been noticed before by any person, that, in the preparation of this acid, the receiver always contains, besides phlogisticated nitrous acid, a little vinegar*.

I likewise tried whether the *saponaceous extract of urine*† would yield any saccharine acid. I found in it, however, no traces of that acid, but instead of it, I got a salt, which, after it was purified, agreed in every respect with the salt of benzoin. But, that

* At the time that Scheele wrote this, he could not possibly know that Mr. Westrumb had already particularly pointed out the same circumstance. They are both, therefore, equally entitled to the honour of this important discovery. CRELL.

† When urine is evaporated to the consistence of honey, and is afterwards digested in spirit of wine, most of the salts which the urine holds in solution, separate from it. This spirituous solution is afterwards to be evaporated to the thickness of an extract. Now, as this extract is soluble both in water and spirit of wine, it may be, as it has been by Mr. Rouelle (*Journal de Medecine, Novembre, 1773*) properly enough termed saponaceous extract of urine. Mr. R. says that it contains a good deal of volatile alkali; but he does not tell us with what acid this alkali is combined.

this salt was not generated during the process (viz. during the digestion with nitrous acid) I concluded from hence, that, on dissolving this extract in a little water, and adding to it vitriolic or marine acid, the solution was immediately thickened by the separation of the same salt. This salt, or this acid, is combined, in the extract of urine, with volatile alkali, and hence it is in the form of a sal ammoniac, which is soluble in spirit of wine. I have remarked, in a former paper, that the acid of sugar of milk yields, in like manner, when distilled, a salt of benzoin. This vegetable acid therefore, does not, it seems, undergo any change during its circulation with the blood.

REMARK. No acid seems to come nearer to the nature of the acid of apples, than the acid of milk. The only difference between them seems to consist in this, that the compound of calcareous earth and acid of milk is soluble in spirit of wine, whereas the compound of calcareous earth and acid of apples is not. Probably this differ-

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ence

ence in the acid of milk is owing to the fermentation of the whey.

II. ON THE MEANS OF CONCENTRATING VINEGAR;

By Mr. LOWITZ.

(Continued)

§. XVII.

IN the further prosecution of the experiments with concentrated glacial vinegar, many other curious phenomena were observed:

If vinegar concentrated by freezing (*al-kobol aceti*) be distilled very slowly, and without any addition, there comes over, at last, a small quantity of crystallizable vinegar, from which, by means of a great degree of cold, a little glacial vinegar may be procured.

procured. This method, however, is not only very tedious, but the vinegar thus obtained, is highly phlogisticated, and empyreumatic.

§. XVIII.

By abstracting a large quantity of common distilled vinegar; over charcoal powder, first in a water-bath, and afterwards in a sand-bath, crystallizable vinegar may in like manner be obtained; but, on account of the great proportion of water in distilled vinegar, this method is extremely tedious.

§. XIX.

Reflecting on this property of glacial vinegar, viz. that it requires for its liquefaction, a degree of warmth, considerably greater than that in which the completely liquefied glacial vinegar is capable of crystallizing; I was led to discover a method, by which crystallizable vinegar may be made to crystallize in a cold, 15 degrees less than that which was before required, viz. Into some previously liquefied glacial vinegar, I dipped

a thin cotton-wick, crystallized the vinegar by placing it in snow, and then in a cold of 158 degrees, applied the same wick, with the crystals adhering to it, to the surface of some crystallizable vinegar, of the strength of 38 degrees; there immediately formed round the end of the wick a number of needle-shaped crystals, which visibly increased in size; the surface of the vinegar became covered over with an icy crust; a great number of radiated crystals, exactly resembling flakes of snow, gradually sunk to the bottom of the bottle, where they continued to accumulate, and at length the vinegar, throughout, shot into fine large crystals. By this mode of treatment, it is rendered unnecessary to wait for a cold of 173 degrees, so that we have it in our power at all times, to obtain vinegar in the state of very beautiful transparent crystals, of a regular prismatic shape, and several inches in length.

§. XX.

From two pounds three ounces of perfectly dry *soda acetata*, and a pound and a half
of

of highly concentrated oil of vitriol, I prepared, according to Mr. Westendorf's method, 13 ounces of an alkohol aceti, of the strength of 32 degrees. By exposing it in the night time to a cold of 174 degrees, and by applying to its surface, some crystals of glacial vinegar, adhering to a cotton-wick, I immediately brought it to crystallize; and the next morning, in a cold of 182 degrees, I found it formed into beautiful prismatic crystals, which were three inches in length. After the mother-ley was poured off, these crystals weighed three ounces, two drams and a half. I distilled this mother-ley, the strength of which still amounted to 24 degrees, with two pounds of charcoal-powder, in a water-bath; the vinegar which came over into the receiver, had a very sweet smell, and was now only of the strength of 16 degrees. From the residuous powder, I afterwards obtained by distillation in a sand-bath, two ounces, six drams and a half, of a smoking crystallizable vinegar, of the strength of 36 degrees.

§. XXI.

The properties of this glacial vinegar, prepared from Mr. Westendorf's vinegar, are precisely the same as those of the glacial vinegar, which is obtained by means of charcoal powder alone (§. II) whence it is evident, that the vinegar is not in the least altered in its nature by its combination with a foreign body (viz. the alkali) and its subsequent separation from it.

§. XXII.

Last year, on the 28th of February, after much reflection, I was so happy as to find out another very effectual method of separating the acetous acid from the other substances combined with it, so as to obtain it at once, in the state of a glacial vinegar of the greatest possible strength. The separating medium which I thought of, is a vitriolated tartar supersaturated with vitriolic acid, a salt, in which, conformably to my purpose, the vitriolic acid exists in a perfectly dry and dephlegmated state.

§. XXIII.

§. XXIII.

My first business was to contrive an easy method of preparing this salt, whose properties have been hitherto but little examined; after various trials, I hit upon the following successful process:

Mix together in a tall matrafs seven parts of water with an equal quantity of oil of vitriol, and to the very hot mixture add, as quickly as the effervescence will permit, four parts of salt of tartar, or levigated pot-ashes. As soon as the mixture becomes cool, the supersaturated vitriolated tartar shoots into fine large crystals. After the whole is become quite cold, and the crystallization is at an end, the liquor (which may serve again for a fresh mixture of the same kind) is to be poured off, and the salt which remains in the matrafs is to be shaken together, and to be well rinsed, as quickly as possible, with cold water, in order to cleanse it from all the vitriolic acid which adheres to its surfaces, and which would otherwise be prejudicial to the dry acid. The crystals are afterwards
to

to be dried by exposure to the fire, and to be triturated to a very fine powder, which, just before it is used, must be again thoroughly exsiccated

§. XXIV.

By means of this salt, a highly concentrated glacial vinegar may be obtained in the following manner :

Let three parts of acetated soda, prepared with vinegar distilled over charcoal, and evaporated to perfect dryness, be melted in a strong heat ; then pour it out, and rub it to a very fine powder. Mix this powder very accurately with eight parts of super-saturated vitriolated tartar (§. 23.) that has been previously well dried, and in like manner reduced to a fine powder, put the whole into a retort, and distil with a gentle heat ; in such manner, that, along with the drops some vapours also may be perceived to come out of the neck of the retort ; but by no means so that the receiver shall be filled with these vapours. Notwithstanding the moderate heat, the vinegar comes over very fast,

fast, and the quantity of glacial vinegar, of the strength of 54 degrees, which is thus obtained, amounts to nearly two parts, and possesses all the characters which have been before (§. 6.) described.

§. XXV.

By this process, seven pounds of glacial vinegar may be obtained from 300 pounds of common vinegar; and from five pounds of distilled vinegar, of the strength of five degrees, two ounces of glacial vinegar may be procured in the space of six hours.

§. XXVI.

This glacial vinegar generally acquires an unpleasant smell, from which, however, it may be completely freed by distillation with charcoal powder, in the proportion of at least five or six parts of the latter to one part of the vinegar; or, to three ounces of this vinegar add about a dram of perfectly dry and finely pulverized acetated-calcareous earth, shake them well and repeatedly together,

ther, and let the mixture remain exposed to the sun-shine, till the disagreeable smell is entirely gone; then re-distil the vinegar in a gentle heat.

§. XXVII.

In its purification, whatever be the way in which it is effected, this glacial vinegar is unavoidably lowered some degrees in strength. The reason of this will be assigned at §. 34. and 35.

§. XXVIII.

The melting of the acetated soda (§. 24.) only serves for the expulsion of all the watery parts from that salt; but a glacial vinegar of the strength of between 46 and 50 degrees, may be procured from a merely exsiccated soda acetata.

§. XXIX.

For the production of glacial vinegar, by means of the supersaturated vitriolated tartar, not only the acetated mineral alkali,
but

but also well dried acetated calcareous earth may be employed.

§. XXX.

How much preferable this last process, with vitriolated tartar saturated with an excess of acid (§. 24.) is to the other (§. 20.) in which glacial vinegar is prepared from Mr. Westendorf's vinegar, will appear from a comparative statement of the following particulars :

(1) In the direct separation by means of oil of vitriol, we are restrained from adding to the acetated salt as much of the vitriolic acid as is necessary for the expulsion of all the vinegar contained in the acetated soda, lest the required acetous acid should be too much debased by vitriolic or sulphureous acid : whereas, in the separation by means of the supersaturated vitriolated tartar, no harm at all can arise from adding an excess of the vitriolated salt, for the purpose of dislodging the whole of the vinegar contained in the acetated salt ; for the superfluous portion of vitriolic acid, combined with the
alkali,

alkali, adheres to it too strongly, and is too fixed in the fire, to be disengaged and raised up by that gentle degree of heat, which is sufficient for the separation of the acetous acid.

(2) In the affusion of a very concentrated oil of vitriol upon the thoroughly dried acetated salt, we are far from being able to make the vitriolic acid penetrate the salt in an equal manner, which, however, is a matter of great consequence: whereas, in the trituration of the acetated salt with my supersaturated vitriolated tartar, the most equal commixture of both salts is readily effected.

(3) During the affusion (though it be performed with the greatest caution) of highly concentrated oil of vitriol, a great heat is excited, whereby some of the vinegar is instantly consumed, a volatile sulphureous acid is produced, and part of the acetous acid is dissipated and lost in the form of vapour. But nothing of this kind takes place in my last invented method; for there
the

the salts do not begin to act reciprocally upon each other till the fire is applied.

(4) The vinegar prepared according to Mr. Westendorf's method, always contains an admixture of vitriolic acid, which, however, for the reason assigned in No. I. is by no means the case with my glacial vinegar, provided the management of the fire is properly attended to.

(5) Twenty-four ounces of acetated soda, treated with oil of vitriol, give only 12 ounces of a barely crystallizable vinegar, of the strength of 32 degrees, from which, at most, only six ounces of glacial vinegar can be procured. On the other hand, the same quantity of acetated soda (previously fused) treated with the supersaturated vitriolated tartar, yields nearly 16 ounces of glacial vinegar, of the strength of 54 degrees.

(6) Glacial vinegar cannot be obtained from Mr. Westendorf's vinegar, except in winter, and by exposure to a very intense cold: but in my last described method, an exceedingly strong glacial vinegar may be immediately prepared at any time.

(7) The

(7) The supersaturated vitriolated tartar may be prepared from various pharmaceutical residua; for instance, from the vitriolated tartar which remains after the purification of pot-ashes, and the residuum from the liquor anodynus; and even the residuum after the preparation of glacial vinegar by means of supersaturated vitriolated tartar, may, when the Glauber's salt which is produced is separated from it, be made to serve again, on the addition of fresh oil of vitriol, for another similar process.

§ XXXI.

A weak glacial vinegar may be concentrated in the following easy, and at the same time, entertaining manner:

Place the bottle containing the fluid glacial vinegar, which is to be concentrated, up to its neck in a vessel full of ice and water: in another quantity of previously concentrated glacial vinegar, surrounded in like manner with ice, dip a thin cotton-twist or wick; as soon as some crystals are perceived to have attached themselves to the wick, apply

apply the same to the surface of the glacial vinegar, which is to be concentrated, and it will be seen to shoot into considerably large crystals. In the course of an hour or two, let the weaker part which still remains fluid, be poured off, into another bottle, from the concentrated and crystallized glacial vinegar.

§. XXXII.

In winter this concentration of the glacial vinegar (§. 31.) may be effected in the following still more simple manner:

Pour the glacial vinegar, which is to be concentrated, into a glass bottle or jar, with four sides; crystallize the vinegar by placing it in snow, or by exposure, in any other way, to cold; and then place it in a warm room, before the window (where, however, the rays of the sun are prevented from entering) in such manner, that one side of the bottle may come into close contact with the cold pane of the window. In the course of ten or twelve hours, the strongest part of the acid will arrange itself, in clusters of crystals,

all along that side of the bottle which touches the pane of the window, whilst on the other side of the bottle which faces the room, the much weaker portion of the vinegar is found in a perfectly fluid state; so that all that remains to be done, is to pour off this fluid part into another bottle.

§. XXXIII.

From the weaker portion that is separated in the concentration of the glacial vinegar (§. 31. 32.) more glacial vinegar may yet be obtained, by exposure to cold, and more especially by means of the before-described (§. 19.) process with the cotton wick: and even the still weaker mother-ley, which remains at last, may very easily be brought to crystallize afresh by the help of a gentle distillation, in the manner pointed out at §. 11.

§. XXXIV.

Decisive experiments, and those frequently repeated, have convinced me, that the 54th degree is the highest pitch of concentration

tration to which glacial vinegar is capable of being brought. In spite of all the pains I have taken, I have never yet been able, either by distillation, or by any of the other concentrating methods (§. 31. and 32.) to push the concentration of glacial vinegar even a single degree farther; on the contrary, the vinegar was rendered weaker by some degrees in all such attempts.

§. XXXV.

The reason of this seems to be, that the acetous acid, like some of the mineral acids, when still further dephlegmated, or deprived of its watery parts, is no longer capable of remaining in a condensed state, but probably assumes an aërial or gaseous form; otherwise, in the further abstraction of its watery parts, how could there be such a constant diminution of its strength (§. 34.) evidently owing to a loss of acid?

§. XXXVI.

Upon this principle we can easily account for the extraordinary quantity of air bubbles,

bles, which are produced as often as the glacial vinegar passes from the fluid to the solid crystallized state. The white appearance of the crystals depends on the same cause.

§. XXXVII.

Hence, too, we see the impossibility of having a glacial vinegar, which shall retain its solid crystallized form during summer, unless it be kept in a place where the warmth never exceeds, at most, 126 degrees. Thus, for example, in a cellar whose temperature remains constantly at 131 degrees, summer and winter, a strong glacial vinegar would never become fluid; but if the same vinegar should be brought into this cellar in a perfectly liquefied state, it would never crystallize there, on account of the difference between the temperature required for the crystallization, and that which is necessary for the liquefaction of glacial vinegar (§. 6. n. 1 and 2.)

§. XXXVIII.

By very accurate experiments I have found, that the least cold which suffices, without

without the application of ice or snow, for the crystallization of a perfectly fluid glacial vinegar of the strength of 54 degrees, is that of 132 degrees. All that is needful is, after letting the glacial vinegar stand some time in water of the above-mentioned temperature, to open the bottle, and shake it a little, so that the external air may enter, and have free access to the vinegar.

§. XXXIX.

Lastly, we may at all times obtain glacial vinegar in a crystallized form, by means of an artificial cold; viz. Pour the vinegar into a thin, slender bottle, wrap round this some rag moistened with a little rectified vitriolic æther, and whirl it about briskly in the air.

§. XL.

It is a circumstance worthy of notice, that the weakest glacial vinegar, in respect to the quantity of alkali necessary to its saturation, is stronger than the strongest smoking spirit of nitre.

III. METHOD OF
 DYING LINEN AND COTTON
 OF A
 BEAUTIFUL DEEP AND LASTING
 BLACK COLOUR,

By Mr. VOGLER, of Weilburg.

THAT the art of dying linen and cotton of a full and permanent black colour, was attended with great difficulty, not only the accounts of the manufacturers and dyers themselves, but also my own experiments on the subject, convinced me. After long and frequent trials, however, I at last discovered a method, which, on every repetition, was found to answer. The following is an exact account of the same.

(1) I took two pints (32 ounces) of pure soft water, rain or snow water for example, such as is collected in the month of February or March, and mixed with it, in a large bottle, two, or two and a half ounces of
 common

common aqua fortis, and threw in, by degrees, of litharge, also, two or two and a half ounces, or even half an ounce more, if the acid happened to be rather strong. The bottle, not too strongly corked, was then put in a warm place, and shaken from time to time.

In a few days, the clear part was poured off from the sediment, into a deep earthen, leaden, or pewter vessel. In this nitrous solution of litharge, the cotton or linen intended to be dyed (such as had been previously well washed, though unbleached) was immersed, and suffered to macerate and soak from ten to twelve hours; at the end of which time, after being three times well rinsed and wrung out in pure cold water, it was dipped, well pressed out, but still wet, in glue-water, of not too weak a quality, and was afterwards dried in the shade, care being taken that the glue-water should be previously pressed, but not washed out.

Raw, unbleached, and loosely spun linen, or cotton yarn, such as had been previously lixiviated, that is, steeped in warm ley, and

afterwards rinsed out in pure cold water, is thereby better prepared, and takes the maceration and colour readier than either the knitted or woven stuff.

Each skain is to be tied up with two loose and broad bindings of tape or string, by means of which, if they should become entangled together, during the soaking or dying, they may be easily separated from one another, and be reduced to order again.

The glue-water, in which the linen or cotton (after the steeping in the solution of litharge) was immersed, is made by boiling the common brown carpenter's glue with a little water to a jelly, of which jelly, so much is afterwards to be dissolved in boiling water, as shall give a glutinous consistence to it when taken between the fingers, and yet not render it so thick as to prevent fluidity. This causes a full and lasting colour, as I have already remarked in a former paper.

(2) I took three parts of an ounce of well-bruised galls, and boiled the same in two full pints (34 or 35 ounces) of clear water, for example, rain or river water. After seven

or

or eight minutes boiling, I put in three parts of an ounce of common salt, and immediately upon its solution, threw in the linen or cotton already prepared with the saturnine solution and glue-water (1). After they had remained boiling about seven or eight minutes, they were taken out, rinsed (and wrung out) three successive times in cold water, and dried in a shady place.

The stuff, which before was white, may now be observed to have acquired a dark grey colour, with a brightish yellow cast; which is to be regarded as a very good basis for a black colour.

In a former paper, I have shewn that the different solutions of lead and its calx will render stuffs so prepared (when boiled with different colouring matters) capable of receiving an extremely full, though very dark and black colour, which penetrates deeply and copiously into the substance of the stuffs, and hence they may be used very advantageously in dyings of brown and black.

In place of galls may be used an equal quantity of oak bark, the thin bark of the
alder

alder-tree, the rind of the walnut-tree root, the flowers or the shells of the pomegranate, *rhus coriaria*, *potentilla argentea*, or the root of tormentil,

The common salt, in like manner, contributes its share towards saturating and fixing the colour, and it is best to add it towards the end of the boiling, in order that the extraction of the colouring part from the galls, &c. as well as the impression of the colour upon the substance of the linen, may not be thereby impeded.

(3) I prepared a solution of three parts of an ounce of common copperas, or vitriol of iron, and the same quantity of common salt, in two pints, or 32 ounces of clean hot water. In this I immersed the stuffs prepared in the manner (2) with galls, or other vegetable astringents, which in consequence had become already, in some measure, black. After eight or ten hours, they were taken out, rinsed, and successively pressed out, in clean fresh water, and dried in the shade.

The advantage derived from the common salt in this solution of copperas is, that not only

only less of the ground-colour with which the linen is already prepared, is thereby extracted, but the dyes, which are to follow, become better impressed, and are more fully saturated, as appears from some of my former experiments.

(4) I boiled three parts of an ounce of Brazil or log wood, in two full pints (34 to 36 ounces) of rain or river water, and added to it, after it had boiled about seven or eight minutes, a quarter of an ounce of white starch, such as had been finely levigated, and previously broken down in half a cupful of fresh water. As soon as this was dissolved by the boiling, I put into the liquor the linen, prepared as in (3) and suffered it to boil about seven or eight minutes; I then took it out, and after rinsing and wringing it out three times in cold water, I dried it in the shade.

If its colour should be too faint, that is, not yet a beautiful and full black, which, however, unless there be some mismanagement, cannot happen, then the stuff must be macerated again in the copperas solution (3)

I

and

and boiled a second time with the Brazil wood.

It will then certainly be of the desired full black colour; which, however, is not yet so fixed as to bear being washed in ley, or soap and water. In order to bring it to this perfection, the linen or cotton was in the last place treated in the following manner:

(5) One ounce of well bruised galls, or any other astringent vegetable substance (mentioned in 2) and two full pints, or 35 ounces of glue water (such as between the fingers discovered a gummy consistence) were placed over the fire. When the mixture had boiled about seven or eight minutes, an ounce of copperas was dissolved in it, and it was taken off. In this decoction, as soon as it was cold, the linen or cotton, already prepared (as in 1, 2, 3, 4) was steeped, and suffered to remain an hour, when it was taken out, and dried in the shade, the liquor having been previously pressed, but not washed, out. In this state the pieces of stuff were suffered to remain untouched for some time, that the dye might, in the same manner

manner as in the printing of cotton and Indian stuffs, eat in and become fixed. At the end of three or four weeks, the stuff must be washed out several times in clean cold water; it ought not to remain unwashed longer, otherwise it may be too much eaten into by the last colour or dye, and become rotten.

The decoction of Brazil wood (4) may be set aside (if the operator chuses) for some weeks, till it has acquired a bad smell, by which it does not lose, in the least, its colouring property, but becomes much more useful.

The addition of white starch promotes remarkably the solution and extraction of the colouring particles from this dye-wood, and helps to give the linen or cotton a more beautiful and deep colour.

For boiling and steeping the colours and stuffs, earthen, brass, or copper vessels are proper, such as are not too large, but the deeper the better, as in such the articles to be dyed may be immersed conveniently, and cannot rise so easily above the surface.

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IV. EX-

IV.

EXPERIMENTS WITH MANGANESE,

AND

PARTICULARLY WITH ITS AERATED CALX;

By Mr. J. J. BINDHEIM, of Moscow.

ON analysing manganese, through all its component parts, I obtained from three ounces of that sort which comes from Ilefeld (previously separated as much as possible from all foreign matter) one dram, 55 grains of siliceous earth, 24 grains of ponderous earth, two drams of calcareous earth, with a small appearance of copper and iron; and three ounces six drams of aerated calx of manganese, were separated from the solution, by means of the mild alkali.

From three ounces of black manganese, a considerable quantity of the purest dephlogisticated air was obtained by means of the pneumatic apparatus.

The

The recomposition or synthesis of the same, from the separated parts, I effected both in the humid and dry way.

(a) To 120 parts of the calx of manganese, combined with fixed air, which are equal to 100 parts uncombined with air, I added eight parts of filiceous earth, one part and a half of aerated ponderous earth, and eight parts of crude calcareous earth. The two last were dissolved in nitrous acid, and then heated a little. With the calx of manganese the treatment was the same.

Both solutions were then poured together, and the filiceous earth, which was finely rubbed down, was thrown therein. I put the whole into a glass vessel over a slow fire, and suffered all the nitrous acid to evaporate, whereby a shining and firm mass was obtained. In order to give this a resemblance to natural manganese, without destroying its shining particles, it was coarsely rubbed down, mixed into a soft paste with water, and dried. The result was in every respect like the natural manganese, which underwent, through all its component parts, a decom-

decomposition, and afterwards from its separated parts a recombination.

(b) The same substances were taken in the same proportion as before, with this difference only, that the calcareous and ponderous earths were deprived of their aerial acid by a strong heat; the aerated manganese was moderately heated by itself, till it became black; these were mixed warm, with eight parts of siliceous earth; and the whole was worked into a paste with a little water, and dried. Internally this artificial compound resembled the natural one, but in its external appearance it differed a little, as it wanted lustre.

One ounce of aerated calx of manganese was dissolved quickly, and with ebullition, in diluted nitrous acid, in consequence of which it lost two drams one scruple in weight. The solution was placed upon an open fire in a glass retort. During the evaporation some shining metallic particles attached themselves to the sides of the retort; and as the mixture acquired consistence, it threw up a strong scum. After the acid was
entirely

entirely evaporated, there were found in the residuum, five drams, two scruples of revived metal. On repeating the experiment with pure dephlogisticated nitrous acid, the result was the same.

I discovered the effects of aerated calx of manganese, upon red precipitate (calx of mercury) by the pneumatic apparatus in the following manner: from a mixture of 40 grains of the first, and 80 grains of the last, a quantity of air was disengaged before the mixture, which was placed in a stove, had well become warm; on applying a very moderate heat, a watery evaporation took place. During the extrication of the air, the quicksilver shewed itself in a revived state, and on increasing the heat, it was completely sublimed and driven over. It amounted to 70 grains in weight. The air which had been collected in this process, and which had nearly filled an eight ounce receiver, proved, on examination, to be of two kinds, viz. fixed air, which was the larger portion, and common air.

It appears, in truth, as if there was some contradiction here, and some fault in the result, while, in this experiment, the red precipitate gave out vital air; however this does not follow, nor can it follow. Ought not then the vital air, which was here found in the mixture, to be considered as the parent of the fixed air, in somewhat of a superabundant quantity, and that the latter owed its origin to the former?

Aerated calx of manganese and white arsenic, of each one dram, were mixed with an equal weight of the alkali of tartar, and first were brought into heat in a firmly covered crucible (otherwise the coverlet will be thrown off). After the elastic fluids were expelled, the crucible well luted, and all was dry, it was placed in a wind-furnace, and by means of a strong fire, the contents were fused in half an hour. I found, on breaking the crucible, 77 grains of a black shining regulus, which did not possess any very strong cohesion. When a bit of it was worked with the blow-pipe upon charcoal, it

it came immediately into fusion, whereby the arsenic was separated. It then lost its easy fusibility, and a hard black shining button remained behind.

In diluted vitriolic, nitrous, and marine acids, the aerated calx of manganese dissolved completely in the cold; with the first it yielded small white shining crystals, which lay one over the other, and were easily soluble in water. With the nitrous acid, similar crystals were obtained, which, by a moderate application of heat, were easily fused; they remained dry in a temperate, but not moist air: the crystals with marine acid, on a moderate exposure to the blow-pipe upon charcoal, suffered the acid presently to fly off, and then assumed a metallic lustre. With the fluid acid of phosphorus (from bones) they produced a sort of gummy mass, which easily became moist in the air, and after a continued blowing upon heated charcoal, melted into a small metallic button.

By the help of heat, and a large quantity of the acid, it was completely dissolved in

Mr. Westendorf's vinegar, and the clear brown solution, afforded flat, pointed, lancet-shaped crystals, which did not deliquesce on exposure to the air. When a little of the aerated calx of manganese, mixed with water saturated with fixed air, was set to freeze, the congelation took place not only faster than happens without the addition of the latter to the former, but the crystallization of the ice was remarkably different.

Nature and operation of the aerated calx of manganese, treated in the humid way with dephlogisticated alkali.

WHEN crystallized phlogisticated vegetable alkali, and an equal quantity of the aerated calx of manganese, with a due proportion of distilled water, were digested together, the solution afterwards filtered, and the crystallization promoted, a pure crystallized alkali was obtained, which no longer possessed any vestige of the blue-colouring property. If this however happened not to be pure previous to phlogistication, or was phlogisticated

phlogisticated with impure Prussian-blue, which, when not sufficientlyedulcorated, is commonly mixed with vitriolated tartar, then this neutral salt is first precipitated, and becomes deprived, like the other, of the blue-colouring matter, which is found in the residuum, along with the calx of manganese therewith employed. By this operation however, the colour itself, and the weight first taken, suffer little or no change.

From this residuum the basis of the blue-colouring matter in the Prussian-blue, appeared in a dry state, and it was obtained from thence in several ways, of which at present I will only relate two.

I divided the residuum into two equal parts, one of which I digested in Westendorf's vinegar, till the acid discovered no more action upon it. A fourth part of it remained undissolved. The other half I treated with diluted nitrous acid in the same manner.—The result was like the foregoing in every particular, and both discovered the following properties : when a little of the undissolved residuum was mixed with a solu-

tion of vitriolated tartar, it was immediately tinged of a yellow colour, and thereby acquired the property of precipitating dissolved iron, blue. The same effect also took place on mixing some of it either with the mild alkali, separated before, or with another in solution: when digested with weak vitriolic acid, which was unrectified, and diluted with a good deal of warm water, it threw down a clear blue precipitate. In like manner, on letting it stand with colourless marine acid in a warm place, it thereupon became of a yellow colour. This did not proceed from iron; on the contrary, the solution of iron was precipitated of a light blue. As one of its peculiar properties, I also remarked that, on holding some of it over a lighted candle, upon a polished knife, it began to inflame quickly, as soon as the iron was well heated, and there remained behind scarcely more than half, that, like iron, was magnetic, a property which it had not before.

From repeated experiments with this extract or quintessence of Prussian-blue, I have
5
found

found that the application of any acid, whether free or fixed, greatly contributes to quicken the operation of the blue colouring matter. The result was the same with fixed air, volatile sulphureous acid, vitriolic, nitrous, marine, and acetous acids; and probably also the other remaining acids, with which I have not yet made any experiments, would have the same effect. Perhaps this colouring substance is also present in many others, though with all the known neutral salts, the case may not be the same as it is here with the vitriolated tartar. I leave this to be determined by future observations.

From the experiments which follow, it appears that the colouring matter of Prussian-blue has a stronger attraction to the aërated calx of manganese, than to the vegetable alkali.

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(This paper will be continued in the next number)

V. EXPERIMENTS
ON THE
ALKALINE SUBSTANCES
USED IN
BLEACHING;

By RICHARD KIRWAN, Esq. F.R.S. and M.R.I.A.*

§. I.

BLEACHING being one of those arts which consist in scarce any thing else than a particular application of some of the general principles of chemistry, it might be expected that the knowledge of the instruments it employs, would keep pace with the progress and improvements of that science to which it is subordinate, and so much the more, as the nature of alkaline substances in general, which are its proper instruments,

* From the Transactions of the Royal Irish Academy for 1789. Dublin, 1790.

has

has been in great measure explained by the celebrated Doctor Black, upwards of thirty years ago ; yet it has so happened, that on a late occasion, when a scarcity of these saline substances, imported from foreign countries, unhappily prevailed in this kingdom, it was seriously questioned, whether their place could be supplied by materials manufactured at home. In the course of this discussion, it evidently appeared, from the contradictory testimonies of many of the principal bleachers, that, however they might excel in that art, when well provided with the instruments they employ, they were but little acquainted with the general agency of the instruments themselves, and their respective powers, or even with the most advantageous and æconomical method of employing them. To elucidate these points by an analysis of some of the different substances employed by bleachers, and by giving a sure method of distinguishing the relative powers of every saline substance they may use, together with an account of the best method of obtaining them, as well as of adapting them to the purpose

purpose of bleaching, is the object of this paper. This task, which I have imposed upon myself solely with a view to the utility of the public, requires no ingenuity, and might have been long ago well executed by many others, if chemistry, which has so many votaries of the highest rank in the most civilized parts of Europe, had been more known and cultivated in this country, which, perhaps, of all others, stands most in need of its assistance.

§. II.

Barilha.

Of this substance there are several sorts made of different plants*, but the best is that formed near Alicant, at a distance from the sea, by the combustion of a plant called by the inhabitants *Barilha*, and described by Jussieu in the Memoirs of the Academy of Paris for 1717, under the name of *Kali hispanicum supinum annuum, sedi foliis brevibus*. It seems to be classed by Linnæus under the

* See Colonel Conyngham's letter, report of the Committee of the House of Commons of Ireland, 1788, p. 87.

pentandria

pentandria digynia, by the name of *Salsola vermiculata frutescens foliis ovatis acutis carnis*, and should carefully be distinguished from the various kinds of *Salicornia* which he ranges under the title of *monandria monogynia*; and also from other plants which he calls *Chenopodia*, which yield an alkali, but less pure than the *Salsola*. These plants being dried to the same degree as hay, are burned in pits nearly as kelp is with us; the ashes and salt run into a greyish blue mass, which is the barilha†. The best sort is here called *Sweet Barilha*.

The sweet barilha which I examined was most obligingly presented to me by Mr. Byrne, an eminent merchant of Dublin. It was of a bluish colour, covered over with a saline powder, exceedingly hard, and had a smart alkaline taste. When broken it looked black in the fractured part, and visibly contained pretty large pieces of charcoal.

To find the proportion of fixed air in this substance; having reduced a quantity of it to fine powder, I poured on an ounce of it

† And by the French *Soude*, as being employed in soldering metals.

a sufficient quantity of marine acid, and found it to lose by the action of this acid 80 grains of its weight, consequently one pound Troy of this substance contains 960 grains of fixed air (mixed with a little that had an hepatic smell) that is, exactly one-sixth of its weight. Other parcels contained somewhat more, and others somewhat less.

As this substance evidently contained some parts that were soluble in water, and some that were insoluble therein; to discover the weight of each, I poured on one pound of it reduced to fine powder, thirteen pounds of water, moderately hot, successively; this water had previously been boiled and filtered, and contained no other impurity than a slight trace of common salt. This quantity of water was necessary to exhaust all the soluble matter in the barilha.

The solutions were taken in six different portions, none of them betrayed the smallest mark of sulphur, of which I was assured by trying them with the nitrous solution of silver, nor did the Prussian alkali discover any vestige of iron.

By

By eighteen successive evaporations and crystallizations, I obtained 4881 grains of saline matter, the different species of which I shall presently mention, and 2903 of insoluble matter.

It may at first sight appear extraordinary, that the saline matter and the insoluble part should weigh more than the pound of barilha that seemed to afford them, for this amounts only to 5760 grains, and the two former weigh 7784 grains; but it should be considered, that these products were obtained not from the barilha alone, but from the barilha and the water in which the salts were dissolved, whose crystals retained a great quantity of it, and also from the air to which the solutions were exposed, and which they absorbed in large proportion.

As the quantity of the insoluble matter was subject to no such deceptive appearance, I began by examining the weight of that, for this being subtracted from 5760 grains, necessarily determined the true weight of the saline part; and as the state in which the saline part exists in barilha depends, in
some

some measure, on the earths and charcoal with which it is united, as well as the most advantageous method of using it, I examined the nature and quantity of these very minutely.

Having, therefore, dried the insoluble matter for a considerable time in a slow heat, until it appeared as dry as the barilha itself, and having found its weight in that state to amount to 2903 grains, or 6,04791 ounces, I took one ounce of it, and drying it in a heat little below redness, found it to lose 38 grains of moisture.

Another ounce of the same residuum being treated with dilute marine acid, lost 125,5 grains of its weight, and this loss expresses the quantity of fixed air contained in it.

Another ounce being calcined in a white heat for about one hour, lost 200 grains of its weight; and on repeating this experiment, I found the loss amount to 199 grains.

Lastly, on the 281 grains which remained after this experiment, I poured dilute marine

rine acid, and found the quantity of fixed air to be 106 grains.

Hence I deduce the weights of the several substances dissipated by the calcination of an ounce of the insoluble residuum of the barilha.

1st, The weight of the fixed air lost was $125,5 - 106 = 18,5$ grains.

2dly, The loss of moisture was 38 grains.

3dly, The loss of the fixed air and moisture amounted together to 56,5 grains. This deducted from the intire loss, that is, from 199 grains, gives the loss arising from the combustion of the charcoal, and consequently its quantity $199 - 56,5 = 142,5$ grains.

I next proceeded to examine the fixed incombustible part that remained after the above calcination. On the 279 grains of this, which remained after the calcination of an ounce of the insoluble part, I poured a quantity of distilled vinegar, whose specific gravity in the temperature of 62° , was 1,008, and digested that residuum therein for sixteen hours in a heat little more than 100° . After edulcoration and desiccation I found the weight

weight of what remained undissolved to amount to 63 grains. Upon this experiment I reasoned thus: 281 grains of a residuum of this sort contained 106 grains of fixed air, therefore the 279 grains, subjected to the vinegar in this experiment, must have contained 105,24, which were dissipated by the action of the acid; there remained, therefore, of mere earth only 173,76; but of these 63 escaped the action of the acid; therefore there were dissolved 110,76. And as distilled vinegar can act only on calcareous and muriatic earth (the barytic being not expected) the 110,76 that were dissolved, must have consisted of either or both of these, and the undissolved 63 grains must have been argillaceous or siliceous.

To determine the first point, I distilled in a glass retort the acetous solution, which was very voluminous, until no more than about four pints remained. During the distillation, some earth was deposited, which, when dried in a red heat, amounted to four grains. This I re-dissolved, and finding it precipitable by caustic volatile alkali, judged it

it to be magnesia. I then took as much of the acetous solution as amounted to one-sixth of the whole, and pouring caustic volatile alkali upon it, obtained nearly three grains, or more exactly 2,83 grains of magnesia precipitated by the alkali. Whence I concluded the whole solution to contain 17 grains, to which adding the four grains deposited, we have the entire quantity of magnesia dissolved by the vinegar = 21 grains, and consequently the remainder of 110,76 grains, namely, 89,76, must have been calcareous earth.

I also examined the quantity of this earth in another manner; to the five-sixths of the acetous solution that remained, I added gradually vitriolic acid, whose specific gravity was 1,463, as long as any precipitation appeared to take place, then pouring off the vinegar, I edulcorated the residuum, and having dried it, found it to weigh 240 grains, and consequently if the whole acetous solution had been used, the resulting selenite would have weighed 288 grains. Now 100 grains of selenite contain 32 of

calcareous earth, therefore 288 grains contain 92,16 grains, which differs inconsiderably from the former determination.

Lastly, the 63 grains which eluded the action of the acetous acid, being digested in spirit of salt, left a residuum of 41,3 grains, which therefore were siliceous; the remainder not being precipitable by the vitriolic acid, was consequently argillaceous earth; hence the quantities of these ingredients in 480 grains of the insoluble part of barilha, were found to be,

	Grains.	And in the whole insoluble part. Grains.
Fixed air	125,5	759
Water -	38	229,82
Charcoal	142,5	861,82
Calcareous earth	89,76	542,86
Muriatic -	21	127
Argillaceous	21,7	131,23
Siliceous -	41,3	249,58
	<hr/> 479,76	<hr/> 2901,31
Error	,24	Error 1,69
	<hr/> 480,00	<hr/> 2903,00
	<hr/>	<hr/>

I now return to the soluble part of the barilha, which necessarily amounted only to 2857 grains, as $5760 - 2903 = 2857$.

In the first place I obtained 4213 grains of pure crystallized mineral alkali; but these crystals are known to contain but one-fifth of real alkaline substance, the remainder of their weight being fixed air and water of crystallization, therefore one pound of barilha contains but 842 grains of pure real alkali.

Besides this, I also obtained 127 grains of a mixture of mineral alkali and common salt, which I could not easily separate, and 346 grains of a mixture of vegetable and mineral alkali, with a small proportion of extractive matter, and some digestive salt, as I believe; this mass constantly attracted moisture. I weighed it hot and dry, but forgot to examine the portion of fixed air it contained; it could not be less, nor much more, than 28 per cent. and therefore this mass contained about 250 grains of mere alkali.

These solutions, and particularly the last portions, afforded also 125 grains of Glauber's salt, and 70 of common salt; but the

Glauber's salt at least did not exist in a crystallized form in the barilha; and as 100 grains of it are reduced to 42 by expelling the water of crystallization, no more than 53 grains of it can be deemed to have pre-existed in the barilha.

These solutions also deposited 20 grains of earth.

Hence the weights of the different ingredients contained in one pound of sweet barilha are as follows :

Fixed air	960	
Charcoal	861,82	
Calcareous earth	542,86	} 1050,67
Muriatic earth	127	
Argillaceous	131,23	
Siliceous	249,58	
Mineral alkali, pure	842	} 1219
Mineral ditto, impure	250	
Mineral ditto, mixed with common salt	127	
Glauber's salt	125	
Common salt	70	
Earth deposited	20	
	<hr/>	
	4306,49	
Water	1453,51	
	<hr/>	
Total	5760,00	

Hence

Hence we see, that the alkaline part of barilha is nearly in a caustic state, for the entire pound of barilha contained but 960 grains of fixed air, and of this quantity we have seen that 759 were contained in the earthy part. Therefore only 201 grains were contained in the saline part. Now 960 grains of this (and the mere alkaline part did not certainly amount to less) require for their saturation at least 700 of fixed air, therefore they wanted at least two-thirds of the quantity requisite to saturate them. And hence bleachers should not use boiling water to extract the saline substance of barilha, for the alkaline part being in a caustic state, dissolves part of the coaly matter with which it is united, which sullies the solution, gives it a dark hue, and afterwards is deposited on the linen, and cannot be separated by acids.

§ III.

Of Dantzic Pearl Ash.

This salt was also sent to me by Mr. Byrne. It is exceeding white, and if not

F 3

exposed

exposed to the air, very hard, and possesses an alkaline taste.

The quantity of fixed air and earth contained in different parcels of this substance is variable; in some ounces I found the quantity of fixed air to amount to 100 grains, in others to 115; and, therefore, at a medium, it may be rated at 107,5 grains, or 1290 grains, in one pound Troy. The earth remaining after the solution of one pound amounted to 20 grains.

One ounce of this substance gradually heated to redness, and kept in that heat for three quarters of an hour, lost 70 grains of its weight; and being then dissolved in spirit of salt, lost 72 grains; therefore the quantity of moisture in one ounce of this substance was $70 - 107,5 - 72 = 34,5$ grains, or 414 in one pound.

Again, after ten evaporations, I procured from one pound of this substance 505 grains of tartar vitriolate, the last portions of which appeared by the test of the nitrous solution of silver, to contain some digestive salt, and also 36 grains of this last containing a portion

tion of tartar vitriolate; about 18 grains of earth were deposited during the evaporations. The remainder of the pound, after all these deductions, must have consisted of pure mere alkali. Hence the ingredients in a pound must have been nearly in the following quantities :

Fixed air	-	1290	
Moisture	-	414	
Tartar vitriolate		505	
Digestive salt and ditto		36	
Earth	-	38	
		<hr/>	
		2283	5760
Mere alkali		3477	—2283
		<hr/>	<hr/>
		5760	* 3477

Disgusted by the tediousness of these experiments, and recollecting that the alkaline part of these salts was that alone with which bleachers had any concern, I bethought myself of an easy practical method of discovering the presence of this principle, and determining its quantity in all substances in

which it exists, either uncombined, or combined only with fixed air or sulphur.

§. IV.

To discover whether any quantity of fixed alkali worth attention exists in any saline compound, dissolve one ounce of it in boiling water, and into this solution let fall a drop of a solution of sublimate corrosive; this will be converted into a brick colour, if an alkali be present, or into a brick colour mixed with yellow, if the substance tried contains lime.

But the substances used by bleachers being always impregnated with an alkali, the above trial is in general superfluous, except for the purpose of detecting lime. The quantity of alkali is therefore what they should chiefly be solicitous to determine, and for this purpose :

1st, Procure a quantity of alum, suppose one pound, reduce it to powder, wash it with cold water, and then put it into a tea-pot, pouring on it three or four times its weight of boiling water.

2^{dly}, Weigh

2dly, Weigh an ounce of the ash or alkaline substance to be tried, powder it, and put it into a Florence flask with one pound of pure water (common water boiled for a quarter of an hour, and afterwards filtered through paper, will answer) if the substance to be examined be of the nature of barilha, or pot-ash; or half a pound of water if it contain but little earthy matter, as pearl-ash; let them boil for a quarter of an hour: when cool let the solution be filtered into another Florence flask.

3dly, This being done, gradually pour the solution of alum hot into the alkaline solution also heated; a precipitation will immediately appear; shake them well together, and let the effervescence, if any, cease before more of the aluminous solution be added; continue the addition of the alum until the mixed liquor, when clear, turns syrup of violets, or paper tinged blue by radishes, or by litmus, red; then pour the liquor and precipitate on a paper filter placed in a glass funnel, the precipitated earth will remain on the filter; pour on this
a pound

a pound or more of hot water gradually, until it passes tasteless; take up the filter, and let the earth dry in it until they separate easily, then put the earth into a cup of Staffordshire ware, place it on hot sand, and dry the earth until it ceases to stick to glass or iron, then pound it, and reduce it to powder in the cup with a glass pestle, and keep it a quarter of an hour in a heat of from 470° to 500° .

4thly, The earth being thus dried, throw it into a Florence flask, and weigh it; then put about one ounce of spirit of salt into another flask, and place this in the same scale as the earth, and counterbalance both in the opposite scale: this being done, pour the spirit of salt gradually into the flask that contains the earth; and when all effervescence is over (if there be any) blow into the flask, and observe what weight must be added to the scale containing the flasks, to restore the equilibrium; subtract this weight from that of the earth, the remainder is a weight exactly *proportioned* to the weight of mere alkali of that particular species which
is

is contained in one ounce of the substance examined ; all beside is superfluous matter.

I have said that alkalies of the *same species* may thus be directly compared, because alkalies of *different* species cannot but require the intervention of another proportion ; and the reason is, because *equal* quantities of alkalies of different species precipitate *unequal* quantities of earth of alum. Thus 100 parts by weight of mere *vegetable* alkali, precipitate 78 of earth of alum ; but 100 parts of *mineral* alkali precipitate 170,8 parts of that earth. Therefore the precipitation of 78 parts of earth of alum by vegetable alkali, denotes as much of this as the precipitation of 170,8 of that earth by the mineral alkali denotes of the mineral alkali. Hence the quantities of alkali in all the different species of pot-ashes, pearl-ashes, weed or wood-ashes, may be immediately compared by the above test, as they all contain the vegetable alkali, and the different kinds of kelp or kelps manufactured in different places, and the different sorts of barilha, may thus be compared, because they all contain

contain the mineral alkali; but kelps and pot-ashes, as they contain different sorts of alkali, can only be compared together by means of the proportion above indicated.

The application of this test is founded on the following principles:

1st, That a hot solution of a free alkali, or of an alkali combined only with fixed air or sulphur, can hold no *terreno* or *metallico neutral* salt in solution; though it may *alkalino neutral* salt or quicklime, if the alkali be free from fixed air.

2d, That earth of alum cannot be precipitated either totally or partially by the hot solutions of any *alkalino neutral* salt, and therefore that its precipitation is always due to the presence of a free alkali, or at least of an alkali combined only with fixed air or sulphur, to whose quantity it is always proportional. It is true quick-lime will also decompose alum, but the presence of quicklime is easily discovered by the addition of a few drops of any mild alkaline solution, and by the same means as easily separated.

3d, That

3d, That if the earth of alum takes up fixed air (which would increase its weight) this air will be separated by the heat employed in drying it, or at least by the spirit of salt poured upon it, and so may also another heterogeneity which will hereafter be mentioned.

I can see but one inaccuracy attending this test, and that of little moment; it is this, if the alkali contains sulphur, this will also be precipitated with the earth of alum, and increase its weight. The limits of this inaccuracy, at least in common cases, scarcely reach two or three grains, as we shall presently find.

Sulphur is easily detected in any alkaline solution by saturating it with an acid; hepatic air is generally developed, and the liquor becomes troubled.

Not only the proportion, but also the *absolute* weight of alkali in different alkaline substances or ashes may be found by this test, as will appear by the following experiments.

§. V.

Of the quantity of mere alkali in different alkaline substances, as exhibited by the aluminous test.

CrySTALLIZED Soda.

I begin with this as being the purest mineral alkaline substance in a dry form, produced by art. Though it contains only one-fifth of its weight of real alkali, the remainder being water and fixed air, but the proportion of alkali being invariably the same, it is the fittest for a standard with which other substances containing the same sort of alkali may be compared. I found that as much of this substance as would contain 480 grains of mere alkali, would precipitate 725 grains of earth of alum dried and treated as already mentioned, and consequently that 480 grains of *mere* mineral alkali precipitate, 725 of earth of alum.

Note, that in this and all the subsequent experiments, a little more earth of alum is precipitated than is mentioned, because a little

tle

tle always remains in the filtering paper that cannot be had out of it; and I have reason to think by weighing the paper before and after, that this quantity amounts to three or four grains, but as this defect is the same in all cases, it does not invalidate the comparison.

Sweet Barilha.

The solution of one ounce of barilha precipitated 174 grains of earth of alum, therefore, since 725 grains of earth of alum require for their precipitation 480 of mere mineral alkali, 174 grains of that earth require 115,2 of mere mineral alkali. And consequently one ounce of barilha contains but 115,2 of mere alkali; and one pound of barilha should contain 1382,4 grains. This quantity exceeds by about one-eighth the quantity I found by direct analysis, but possibly one pound may contain more than another, for it could scarcely happen that I should commit a mistake of that magnitude.

I must not here omit an odd appearance that occurred in this experiment: The earth of alum in drying acquired a blueish colour,
and

and when spirit of salt was poured on it, to disengage the fixed air it might contain, the blue colour was more developed, and some blue particles floated in the liquor. This seems to proceed from the tinging matter of Prussian-blue, which has been found in barilha. The weight of this I have not examined, but it could not exceed one or two grains.

Cunnamara Kelp.

This was manufactured by Mr. Martin Mealy, and sent to me by Mr. Francis French, an eminent merchant in Dublin. It is a hard porous black substance, mixed with white and grey spots, its smell sulphureous, and its taste mixed, being that of common salt and alkali. One ounce of it dissolved in marine acid, lost 24 grains of its weight, which escaped in an aërial form. This air was hepatic.

Another ounce dissolved in boiling water left an insoluble residue, which being heated in a crucible to redness, weighed 165 grains; this residue effervesced with acids, and seemed for the most part calcareous.

The

The solution by the test of the nitrous solution of silver evidently contained sulphur, and the Prussian alkali gave manifest signs of iron.

This solution precipitated 25 grains of earth of alum, and therefore contained 16,5 grains of mere alkali. During the precipitation of the earth of alum, much hepatic air was emitted, and the earth was sullied by the sulphur, though only a few grains of this can be presumed to be mixed with it.

To find the quantity of sulphur in this kelp, I dissolved two ounces of it in pure water, and saturated the solution with marine acid; the liquor became turbid, and partly by filtration, and partly by spontaneous deposition (for some of the sulphur passed through the filter) I obtained eight grains of sulphur, which gives four grains for each ounce, besides what exhaled in hepatic air.

In order to estimate the quantity of sulphur which a given quantity of mineral alkali is capable of containing, I dissolved 400 grains of crystallized mineral alkali, in fix

times its weight of water (this quantity of the crystals contained 80 grains of mere alkali) and to this I added 80 grains of sulphur, and boiled them for half an hour; only 60 grains of sulphur were dissolved, by which I found that this alkali can take up nearly three-fourths of its weight of sulphur in the moist way; I say nearly, as some earth remained with the undissolved sulphur. With this saturated liver of sulphur, I precipitated a solution of alum, and found the precipitate to amount to 130 grains. Now 80 grains of mere mineral alkali can precipitate only 120,8 grains of earth of alum; therefore nine grains of the above precipitate were sulphur. Yet this small proportion of sulphur was very visible in the earth of alum when heated to 500 degrees, by its strong yellow colour; therefore in the precipitation of the earth of alum by kelp, in which no sulphur was visible, the proportion was incomparably smaller, and no deduction need be made on that account.

There are three methods of desulphurating kelp, or any other alkaline sulphureous compound:

compound: the first is by calcining it in an open fire, by exposing it to a rapid blast of air; and for this a very ingenious contrivance was devised by my much respected friend, Mr. William Dean. The only inconvenience attending it is, that much of the sulphur will be converted into vitriolic acid, and thus combine with the alkali. The second is by saturating it with a vegetable acid, and afterwards calcining it, by which means the vegetable acid will be decomposed; if this method could be cheaply executed, it would be the best. The third is by saturating a solution of kelp with fixed air: this I have endeavoured to effect by putting a solution of two ounces of kelp, into Doctor Nooth's machine for impregnating water with fixed air; the liquor soon became turbid, and emitted a strong hepatic smell; after the sulphur had subsided, I drew off the liquor, and with one half of it precipitated a solution of alum. No hepatic smell was now perceptible, and the precipitate amounted to 40 grains. I dare not say

that this great increase of power in the alkali, was entirely owing to the desulphuration, but some part undoubtedly was: yet the quantity of sulphur I could collect was very inconsiderable, and mixed with coal dust. Kelp may also be desulphurated by nitre, as shall hereafter be shewn. According to Doctor Watson, 30 ounces of kelp afforded him 12 ounces of crystallized mineral alkali, consequently one ounce would afford $\frac{1}{4}$ of an ounce, that is 192 grains, of which one-fifth, that is $= 38$ grains must have been mere alkali.

His kelp might have been better than that I used; but it is impossible that his alkali was pure, as mineral alkali, when mixed with such a quantity of common salt as is in kelp, can never be thoroughly separated from it, but by processes which he certainly did not use, namely, by precipitating a solution of silver in spirit of nitre; estimating the quantity of luna cornea, and afterwards decomposing the cubic nitre; or by saturating the alkali with distilled vinegar, and dissolving

dissolving the neutral salt thus formed in spirit of wine, which leaves the common salt behind.

Strangford Kelp.

This was sent to me by my worthy friend Mr. Braughall. It was much denser, less porous, and in appearance approached more to that of a vitrified mass than Cunnamara kelp; it was at least equally sulphureous. The solution of one ounce of it precipitated only nine grains of earth of alum, and this earth was much more discoloured than that precipitated by Cunnamara kelp. The insoluble residuum of an ounce amounted to 174 grains.

Vegetable Alkali.

I found that 480 grains of the purest and driest salt of tartar (making allowance for the quantity of fixed air it contained) precipitated 331,5 grains of earth of alum.

Dantzic Pearl Ash.

The solution of one ounce of this salt precipitated in one experiment 200 grains of

earth of alum, and in another 220 grains; at a medium 210 grains. Then if 331,5 grains of this precipitate require 480 grains of mere vegetable alkali, 210 grains of this precipitate require 304; therefore at a medium, an ounce of this substance contains 304 grains of mere alkali, and a pound contains 3648. By my analysis it contained 3477 grains; the difference is 171 grains.

We may now determine which of two or more saline substances, one possessing the mineral, the other the vegetable alkali, is best in its kind; for that substance is best in its kind, which approaches most to its proper standard; 725, that is, the precipitation of 725 grains of alum being the standard of the goodness of an ounce of a substance containing the mineral alkali, and 331,5 being the standard of the richness of an ounce of a substance containing the vegetable alkali. Thus, if we compare barilha, and Dantzic pearl ash, as the standard of barilha is to the quantity of earth of alum, an ounce of it precipitates, so is the standard of Dantzic salt to the quantity an ounce of

of

of it precipitates; or $725 : 174 :: 331,5 : 79,5$, by which we see that an ounce of Dantzic salt that would precipitate 79,5 grains of earth of alum, would be as good in its kind as an ounce of barilha that precipitates 174; therefore, since an ounce of Dantzic salt precipitates 210, it is richer in its kind by the difference between 79,5 and 210.

With respect to antacid powers, the mineral alkali is stronger than an equal quantity of the vegetable, that is, will saturate more acid, nearly in the proportion of 48 to 22; yet it attracts acids less, that is, with less force and activity, since the vegetable will take them from the mineral alkali.

But if the quantities of real alkali be unequal, we may compare their antacid powers in this manner: As the precipitate by an ounce of a substance containing the mineral alkali is to 48, so is the precipitate by an ounce of a substance containing the vegetable alkali to a number expressing its comparative antacid power. Thus, with respect to barilha and Dantzic salt, as $174 : 48 :: 210 :$

G 4

58 near-

58 nearly; therefore the antacid power of Dantzic salt is greater than that of barilha, when taken in equal quantities in the ratio of 58 to 48.

Cashup.

The best sort, namely, that marked with the cross arrows, is of a bluish grey colour, exceeding hard, and of a semi-vitrified appearance, its smell sulphureous, its taste scarcely alkaline, and does not attract the moisture of the air. With marine acid, one ounce of it afforded 31 grains of hepatic air. When dissolved in water the residuum of an ounce was 357 grains of a grey earth that appeared to be calcareous for the most part. The solution itself was of a yellow colour, and strongly sulphureous. With the solution of alum it did not effervesce strongly until a good deal was added. The precipitate was of a dirty white, and amounted to 66 grains, of which two appeared to be sulphur. Hence its quantity of vegetable alkali is nearly 93 grains per ounce.

Mr.

Mr. Clarke's Refined Asb.

This I obtained from Mr. Clarke himself. It is of a yellowish white colour, with greenish spots; many pieces are externally white and internally green; it is moderately hard, of a very sharp taste, and effervesces with acids.

An ounce of this substance dissolved in twelve ounces of boiling water, did not effervesce with acids, but precipitated the solution of sublimated corrosive yellow and red as lime water does, and left a residuum of 17 grains, which was evidently calcareous earth. A solution of two ounces of this substance being impregnated with fixed air in Dr. Nooth's machine, deposited five grains of mild calcareous earth; but a solution made in three or four times its weight of water, or without the assistance of heat, contained no lime, and effervesced slightly with acids; and when this salt is some time exposed to the air, its solution contains no lime.

A solution of one ounce of this salt precipitated 89 grains of earth of alum, and there-

therefore contained 129 grains of mere vegetable alkali, to which, if we add 17 grains of insoluble earth, we shall find that the remainder of the ounce, viz. 334 grains, consisted of neutral salts, viz. digestive salt, and perhaps tartar vitriolate, in small quantity. To prove the existence of these, I saturated an ounce of the solution of this refined ash with the nitrous acid, and then dropped into it the nitrous solution of silver; this latter was immediately precipitated in a curdy form, which, as the alkaline part was saturated, could proceed only from the marine acid contained in the digestive salt.

In the report of the Committee of the Irish House of Commons, dated April 1788, Mr. Clarke delivers an account of his method of manufacturing this salt. He mixes five parts weed or wood ashes with one part of quicklime, and suffers them to lie together in a heap for six, nine, or twelve months, and then extracts a ley from them, which he evaporates to dryness. By suffering the lime and ashes to stand together for so many months, he imagines that the common

mon salt contained in the ashes is decomposed, and the quantity of alkali thus increased; but though it is possible to decompose common salt by quicklime, as Mr. Scheele has shewn, yet this decomposition is effected by a very different management; and if in the first part of Mr. Clarke's process such a decomposition were obtained, a recombination would speedily be effected in the second part of his process; for supposing the marine acid to quit its alkaline basis, and to unite with the lime, yet when the alkaline salt and marine selenite are both drawn off into the ley, the alkali immediately decomposes the marine selenite, and re-unites with its acid, according to the well known laws of chemical affinity. So that by this long *maceration* (as he calls it) no advantage whatsoever is gained. However, Mr. Clarke's salt is undoubtedly a valuable preparation for the purpose of bleaching, and may be obtained in a space of time incomparably shorter than he requires.

The neutral salts contained in the solution of Mr. Clarke's refined ash, do not proceed

ceed from any error in his process, but from the bad quality of the ashes he employs.

Common Irish Weed Ashes.

I obtained a parcel of these ashes from Mr. Clarke; it was of a loose texture, dark grey colour, and salt taste, mixed with charcoal, brick-dust, and other impurities. I chose the cleanest, and sifted it. One ounce of it lost by gentle drying 47 grains, and in a red heat 72 grains more.

Twelve ounces of the undried ashes being lixiviated, left a residuum, which, when dried, weighed 4214 grains; the solution was reddish, replete with extractive matter; it afforded a large quantity of digestive salt, and some tartar vitriolate, and very little alkali.

Two ounces of the same ashes being gently heated to a slight degree of redness, lost 186 grains of their weight. One ounce of this calcined ash being boiled in six ounces of water, left a residuum of 344 grains, and consequently contained 136 grains of saline matter; but of this saline matter only 22,4 grains

grains were pure alkali; for the solution precipitated only 15,5 grains of earth of alum; an hepatic smell was perceived during the precipitation of the alum, and the earth was of a dirty colour.

I tried also another sort of ashes which I had from a chandler; it was of a whiter colour and cleaner. The solution of an ounce of it in six ounces of water precipitated only 5,5 grains of earth of alum, and therefore contained but eight grains of mere alkali.

There is a remarkable circumstance attending these ashes, viz. that if they be much calcined, they seem to lose their alkaline properties, and the solution no longer precipitates that of sublimate corrosive reddish, as alkalies not thoroughly aerated do. What this circumstance depends on, I have not as yet examined; but am almost certain it proceeds from the presence of common salt, as fixed alkalies and common salt melt very easily, and thus unite to the earths.

To estimate the goodness of different ashes, some have recommended the use of an hydrometer,

drometer, whereby to discover the strength of solutions of equal weights of these ashes in equal quantities of water ; but as this instrument is equally affected by the presence of neutral salts, as of alkali, it becomes useless.

Table of the quantity of mere alkali in 100 Avoirdupois pounds of the following substances, by the aluminous test.

One hundred lbs.	Mineral Alkali.
CrySTALLIZED soda - -	20 lbs.
Sweet barilha - -	24
Mealy's cunnamara kelp -	3,437
Do. desulphurated by fixed air	4,457
Strangford kelp - -	1,25

One hundred lbs.	Vegetable Alkali.
Dantzic pearl ash -	63,33 lbs.
Clarke's refined ash -	26,875
Cashup - -	19,376
Common raw Irish weed-ash	1,666
Do. slightly calcined -	4,666

(The remainder of this paper in the next Number.)

VI. EXPERIMENTS

WITH

DEPHLOGISTICATED MARINE ACID;

By J. C. SCHMEISSER, of Hamburgh.

THE remarkable appearances produced by the dephlogisticated marine acid air, induced me to go through the whole series of experiments with the same, and to ascertain their results. It was very flattering to me to find that many of my remarks had been confirmed by a Scheele, Scopoli, and by Dr. Hermbstadt in his excellent treatise on this subject. However, some of my observations turned out differently, and give me occasion to object to many conclusions which others have made. These experiments I have already in part repeated, and considered with attention. Some of them I intend to repeat even again, and shall publish their results as soon as I shall have obtained

obtained the vessels which I particularly stand in need of, in order to make my experiments with greater accuracy. I hope I shall then be able to speak more explicitly on the origin and composition of the dephlogisticated marine acid air, and that my labours will be such as may merit some attention from the friends of chemistry.

That every kind of manganese cannot be employed for dephlogisticating the marine acid, without a very different result taking place, is proved by the following first experiment, which I must previously relate.

I took, in the first place, one ounce of manganese, poured upon it two ounces of marine acid, the specific gravity of which was 1,150; but I did not perceive thereupon any volatile marine acid smell; nor even when I placed the retort containing this mixture in a sand-heat of 90 degrees of Fahrenheit's thermometer. These, and other experiments of the same kind, in which I did not obtain any vital air from this manganese, proved that it was not proper for dephlogisticating the common marine acid.

I took

I took likewise another sort of manganese, such as was bought for Piedmont manganese; though it was not so red as Mr. Cronstedt has remarked, yet from its external appearance, it seemed to be better suited for my purpose. I put one ounce of it into a retort, and collected from it 40 cubic inches of dephlogisticated air, such as answered my expectations in all its properties. I took three ounces of the same manganese, whose specific gravity was 1,3222, heated it in a crucible in order to free it of its volatile parts, then put it into a retort, and poured upon it three ounces of marine acid, the specific gravity of which was 1,472. In a temperature of 60 degrees by Fahrenheit's thermometer, which was that of the room where I made this mixture, white fumes, that had the smell of dephlogisticated marine acid, were extricated. On heating the same mixture to 90 degrees in a sand-bath, it discharged yellow vapours, which smelled strongly like nitrous air. The vessel in which I collected the dephlogisticated marine acid air, was of white crystal glass, that I

might better observe all the changes. In a short time I discovered, on a sudden, a thick crust formed upon the surface of the water that had settled in the glass after rinsing; it seemed like ice which had suddenly formed itself. I had scarcely moved the glass, when the whole inner surface of the vessel became covered over with small crystals, resembling those of vitriolated tartar: I was disappointed in examining their nature, and effects with different substances, for the crystals presently disappeared—a singular phenomenon!! When my first receiver, which contained 30 cubic inches, was filled with this aeriform dephlogisticated marine acid, I secured it with a stopple and bladder; and then tied on an empty bladder, which previously weighed 108 grains, but, after I had collected ten cubic inches of the air, it weighed 112 grains. I then continued to apply more small receivers, whereupon I increased the heat still further, so that the retort became heated to 248° . The manganese now appeared to be sufficiently phlogisticated, as I concluded from its white colour.

lour. By this time I had driven over two ounces and a half of a white fluid, which had, indeed, the smell of dephlogisticated marine acid, but tasted like vinegar; I therefore considered it as a kind of weak marine acid. The phlogisticated manganese now weighed three ounces three drams and a half, about thirty grains had been lost, which nearly amounted to the weight of the air obtained. I lixiviated this phlogisticated manganese with distilled water, which gave out a clear-red sourish liquor. I suffered one half of it to evaporate gradually, and thus I procured beautiful, reddish, long, spear-shaped crystals of muriated manganese. Upon some of this, which I dried, I poured again a little common marine acid, and subjected the same to distillation. Here I obtained some dephlogisticated marine acid air, which coloured paper stained with litmus, but had no discoverable effect upon gold. This (according to the experiments of Dr. Hermbstadt) might, for the most part, be mere marine acid air.

On precipitating, by means of mild alkali, the remaining part of the reddish liquor, I

H 2

obtained

obtained 180 grains of a white precipitate, which, from not being yellow, appeared to me to contain no portion of iron. Vitriolic acid held it totally dissolved, so that it could not be calcareous earth. On evaporation, bitter salt crystals appeared, which distinctly shewed the nature of this earth. My observations, therefore, in this respect, coincide with Mr. Ilseman's, and thus the existence of the earth of magnesia in manganese is completely proved. That this earth, however, should effect the dephlogistication of the marine acid, many experiments disprove. I did not perceive, on pouring common marine acid upon this earth, any dephlogisticated marine acid smell, as Mr. Hermbstadt did when he poured marine acid upon the precipitate which he obtained: Perhaps, however, a difference in the manganese employed by each of us in our experiments, might be the reason of this.

In the two ounces and a half of the white fluid which I now poured off (after I had expelled the dephlogisticated marine acid air) there was deposited in a few days, a yellow earth, which I at first took to be a fer-

ferruginous earth. It might, however, be the effect of the strong fire upon the much used manganese. The again crystallized muriated manganese tasted sour, and was easily dissolved in water: the crystals effloresced in the air, and took on, when dissolved, a more dark violet-colour.

I now examined my vessel with the dephlogisticated marine acid air, which without being preserved under water, but only placed in a room of the heat of 70 degrees, was found to be very good:

(1) A wax candle burned in it with a very great flame, though the air had not been transmitted through water. Mr. Scopoli's experiments contradict this; but he certainly did not examine the air soon enough, and his air was, besides, too much phlogisticated.

(2) Paper stained with litmus became immediately altered by it.

(3) I had different corks fitting the bottle, to the inner surface of which I stuck in a piece of glass, which I had armed with some gold leaf, in order to see the effects of

the air upon it; it dropped off from the glass like a yellow oil. A piece of cinnabar, which I had fixed to the cork in the same manner became immediately white upon its surface, and upon scraping this off, I found it to be muriated quicksilver.

(The continuation of this paper in the next Number.)

VII. ON THE AMALGAM OF IRON;

By Mr. VOGEL, of Brehna.

I HAVE lately succeeded in some attempts to amalgamate iron in a more easy and perfect manner than that described by Mr. Leonhardi in his first edition of Macquer's Dictionary, Vol. v. p. 738, and Vol. vi. p. 134. The medium by which such
a per-

a perfect and intimate amalgamation of iron may be effected, is alum. I take half an ounce of fine iron-filings, and triturate them well in a mortar for some minutes, with an ounce of alum, till the whole is reduced to a fine powder. I then add to this powder, from an ounce to an ounce and a half of quicksilver, and incorporate them together, by continuing the trituration for a few minutes. I now pour upon this mass two drams of pure water, and rub the whole well together. (It is remarkable, that the alum is almost entirely dissolved by this small quantity of water). After the trituration has been continued about an hour, the amalgamation of the iron will be perceptible; but we must still persist in rubbing the mass till the amalgam becomes sufficiently perfect; more water must then be added, the amalgam must be rinsed clean, and laid upon blotting paper to dry. By the application of a gentle heat, the amalgamation may be effected in a shorter time.

Now if the amalgam of iron, thus prepared, be attentively observed for some time,

an internal fermentation will be seen to take place. It swells and puffs up like leavened dough ; on pressing the finger upon it, a sort of crackling may be perceived ; cracks and small cavities form within it ; and in the course of twenty-four hours it is increased twice as much in size as it was before it was rinsed out with water. By the application of a moderate heat, or by exposure to the sun, this fermentation is made to take place more quickly, and in a more striking manner. On pressing the amalgam close together, the fermentation ceases.

Might not most ores be decomposed by this amalgam of iron ? The sulphur and arsenic would unite with the iron, and the metal with the quicksilver. I have decomposed the *minera argenti vitrea* and the *minera argenti rubra* by this method, as well as orpiment.

Copper, lead, tin, regulus of antimony, and bismuth, may, in like manner, be very easily amalgamated by means of alum : lead is thereby rendered much whiter.

VIII. ON THE
REGULUS OF PLATINA,
AND
OTHER SUBJECTS THEREWITH CONNECTED;

By Baron Ruprecht*.

I Am not at all surprized that Mr. Hjelm, and others, should not have succeeded in their attempts to reduce tungsten, molybdæna, and platina, because these gentlemen might not have been fully informed of the proper method of proceeding, of the degree of heat which is required to be excited and kept up (but which is a work of great labour) and of the *arranging and placing* of the vessels, on which a great deal depends; or, though they had been furnished with ever so accurate a description of the whole process, it is

* Extracted from a letter communicated to Dr. Crell, by Baron Born, whose recent death must be a subject of regret to every lover of mineralogy, metallurgy, and chemistry in general.

easy

easy to conceive they might fail in their repetition of it, since, on account of the fusibility of all the known vessels, the most inexhaustible patience is required, in order to surmount the many difficulties that occur, and to enable us to crown our wishes with success. It is this that will still weary out numbers of the ablest chemists, and, under the failure of their experiments, will induce them to consider what we have asserted as highly paradoxical, and make them disbelieve the reality of our new semi-metals.

The reguli herewith sent, and especially the *Regulus of Platina* may serve, however, to convince such truly learned, it must be confessed, but at the same time too easy and impatient sceptics, what patience, assiduity, and my furnace are capable of effecting. For this last, we only want infusible and indestructible vessels; for even the purest siliceous earth, as well as pure snow-white talc-stone (*Talkstein*) melted, and though not within the focal heat, vitrified in part.

The regulus of platina was fused *per se* in an hour and three-quarters. It weighs six
drams,

drams, is not attracted by the magnet, and certainly no regulus like it was ever before produced, even in the focus of a burning mirror.

The calx of platina used in my experiments for its reduction and fusion, was precipitated by sal ammoniac from its solution in aqua regia: It was put into a crucible closed with a cover made of very white talc-stone, and by being placed out of the focus, I expected it would stand the heat of my forge, during an hour and a half's blowing; but, to my great disappointment, the cover melted (long before the platina) into a tough glass, which hindered the fusion of the reduced platina; however I collected a few small, round, shining reguli.

I afterwards separated the glass carefully from the reduced platina, and subjected the latter to fusion again, but, owing to the carelessness of my assistant in applying the fuel, the great crucible was almost entirely removed out of the focal heat; otherwise the platina distributed into the three smaller crucibles, would have been still
more

more completely fused. When I repeat the experiment again, I will be more attentive to the position of the crucible, and take care to keep the mixture exactly in the focus; by which means I have no doubt I shall obtain still finer reguli.—Your observation that charcoal-powder is infinitely preferable to soot, for such reductions, is very just.

IX. CHEMICAL NEWS.

MR. Westrumb has finished his experiments on the metallisation of the simple earths. With Mr. Klaproth and others (see the first vol. of the Chemical Journal, p. 307) he proves, that such of the obtained reguli as are attracted by the magnet, consist of iron alone, whilst those which are not attracted by it, are composed of phosphoric acid and iron. He has drawn up, and is now printing a detail of his own experiments,

ments, together with a history of the discovery itself, and the different opinions concerning it.

From the account which Mr. Hassenfratz published, some time ago, of the phosphorescent earth of Marmarosch in Hungary, it was considered to be phosphorated calcareous earth; but Mr. Pelletier has lately proved by his analysis of it (*Annales de Chimie* for June 1791) that it differs very evidently from the phosphorated lime of Estremadura, and that it ought to be classed with the fluor spars, or fluorated limes. According to Mr. Pelletier's analysis, 100 grains of the Marmarosch earth contain of

Water	-	-	1
Siliceous earth	-	-	31
Calcareous earth	-	-	21
Argill	-	-	15 $\frac{1}{2}$
Iron	-	-	1
Marine acid	-	-	1
Phosphoric acid	-	-	1
Fluor acid	-	-	28 $\frac{1}{2}$
			<hr/>
			100 grains
			<hr/>

Mr. Sage (*Journal de Physique* for June 1791) has found that copper may be combined with one-fifth of its weight of phosphorus. The compound thus formed is of a steel colour, and takes a good polish. It is harder than iron, and full as hard as the composition used for making mirrors. It wants ductility. It does not tarnish by exposure to the air; for some of it, after being kept more than a twelvemonth, was not in the least altered in its polish or colour. Mr. Sage obtains his *phosphorated copper* by mixing 200 grains of copper with an equal quantity of glass of phosphorus, and 24 grains of charcoal powder. The mixture is then put into a crucible, covered over with a thin layer of charcoal powder, and exposed to a strong heat. In this process, a part of the phosphorus burns away; but the copper, nevertheless, absorbs about one-fifth of its weight.

Mr. Savarefi (*Annales de Chimie* for July 1791) has, by a further prosecution of his experiments, confirmed the opinion which he some time since (*Chemical Journal* vol. I.

p. 203)

p. 203) threw out, that, in the pretended metallisation of the simple earths, the obtained reguli are, if unattracted by the magnet, siderite; if attracted, siderite joined with a little iron foreign to its composition.

Mr. Sage (*Journal de Physique* for July 1791) has analysed a sulphurated and arsenical ore of cobalt, covered with a reddish efflorescence of vitriol of cobalt. This ore comes from the valley of Giston, in the Spanish Pyrenees. By distilling it with two parts of concentrated vitriolic acid, the sulphur and arsenic are separated from the cobalt. In the beginning of the distillation, there comes over some volatile sulphureous vitriolic acid, then some yellow sulphur; and on urging the fire, the arsenic is sublimed in the form of a white crystalline calx. The sulphur, which is melted and moulded in the neck of the retort, is easily separated from the arsenical calx with which it is covered. In this experiment, a quintal of this ore of cobalt yielded 36lbs. of white calx of arsenic, and 15lbs. of sulphur. The
residuum

residuum contains calcined vitriol of cobalt, of a pale red colour. The arsenic in this ore of cobalt exists naturally in a metallic form; but by distillation with vitriolic acid, it is separated in the state of a calx.

The same number of the last mentioned Journal contains an account of Mr. William Gregor's experiments on a magnetic sand, which is found in the valley of Menakan, in Cornwall, and which he has accordingly named *Menakanite*.

According to some late experiments made, in the small way, by Mr. Schrader, of Berlin (*Crell*, No. IV. 1791) wax bleached by the dephlogisticated marine acid, is not equal to that which is bleached by the common method.

ERRATA in the last Number.

P. 279, last line, for *ground white*, read *ground fine*.

P. 283, l. 11, for *manganese*, read *molybdæna*.

X. EXPLANATORY REMARKS
ON SOME
EXPERIMENTS RELATIVE TO THE NATURE OF
QUICKLIME;

By Mr. SCHEELE.

IN the last number of the Newest Discoveries in Chemistry (1785) I find that Mr. Weber has endeavoured to refute the observations which I made upon his new theory respecting the nature of lime. At first I was not inclined to take any notice of these objections; but on reflecting that there are some persons who are not immediately capable of forming a proper judgment on the subject, and who might therefore join with Mr. Weber in condemning me, I think it necessary to make a few remarks in reply. For, that Black's doctrine concerning fixed air is established as well by old, as by totally
Vol. II. No. 2. I new

new experiments, and that it may, in every respect, be received as the true doctrine, surely no good chemist in Europe will pretend to deny ; but that it should have been found requisite to retouch and amend it a little in some parts, is not at all surprizing—for Dr. Black is but a man.

To the triumphant question respecting the caustic volatile alkali in the distillation of secret sal ammoniac, the following may serve as an answer. The attraction of acids to the volatile alkali is very much weakened by the application of a certain quantity of heat: If the acid is of a fixed or almost fixed nature, this uniting power, viz. the attraction, is entirely destroyed. Hence, on the application of such a due degree of heat, the volatile alkali is disengaged from phosphoric, arsenical, and vitriolic sal ammoniac. This volatile alkali then assumes an aërial form, and retains this form as long as it does not come in contact with water ; but if it meets with water, it unites therewith, loses its elasticity, and lets go as much heat as was necessary for

for imparting to it its aëriform quality. Here then we have the caustic spirit of sal ammoniac.—We know that steam or vapour absorbs or takes up a great deal of heat, and that when a portion of this heat is abstracted from it, water is produced; but this water still retains a considerable quantity of heat in a latent state. The same thing happens in the case of the caustic spirit of sal ammoniac; it still retains heat enough to give it causticity. The mere heat of distillation, which does not exceed that of boiling water, is sufficient to render a mild volatile alkali in some degree caustic; for, a volatile alkali that is completely saturated with fixed air, is quite inodorous; but on being sublimed, it acquires a pungent smell; heat, therefore, separates the two component principles from each other. But as soon as the superfluous heat is abstracted in the head of the glass alembic, the aërial acid and volatile alkali re-unite and form a sal ammoniac, in which the volatile alkali predominates, probably in consequence of the escape of some of the fixed air through the joinings of the apparatus.

Should it be said, that the caustic property which alkaline salts and earths shew after being subjected to the action of the fire, does not depend upon their specific heat; but that they are naturally thus caustic; such an assumption avails nothing: Dr. Black's theory as far as relates to the existence of the fixed air or aërial acid, remains unshaken; and this acid, like all other acids, renders caustic alkaline salts and earths, mild. But of this enough.

As the matter of heat possesses very little gravity, and bears no comparison in point of weight to the aërial acid and water, which calcareous earth loses in the burning; it is a necessary consequence that this calcareous earth is found so light after the burning. If metals contained water and aërial acid, no doubt their gravity would be considerably diminished by calcination; but the contrary is found to be the case. I have now a different conception of this singular phenomenon.

Mr. Weber maintains that the calcareous earth, which he precipitates from lime-water
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by the addition of human urine, effervesces with acids : On the other hand, I have asserted that this precipitate does not effervesce with acids, because it is a pure animal earth. I will here state my reasons for that assertion. Animal earth, such as that which is present in hartshorn, consists of calcareous earth, phosphoric acid, and the acid contained in perluted salt*. This last acid, when treated with phlogiston, yields no phosphorus ; hence the quantity of phosphorus obtained from a given quantity of dry acid of bones, bears no proportion to the weight of this acid. But it has the peculiar property of combining with more calcareous earth than is sufficient for its perfect neutralization ; a property which the phosphoric acid has not. Thus if we saturate this acid of perluted salt with an alkali, fixed or volatile, precipitate lime water with it, and edulcorate the preci-

* At the time Mr. Scheele wrote this, he knew nothing of Mr. Klaproth's experiments on perluted salt (which prove that the acid of this salt is phosphoric acid) but he was afterwards convinced by his own experiments of the truth of Mr. Klaproth's discovery.

CRELL.

pitae well, such a precipitate will not effervesce with acids: But, if we pour some filtrated lime-water upon it, cork up the mixture well, and let it stand for a couple of hours in a warm place, the lime-water will be found to have lost all its taste. If this is decanted and fresh lime-water added, this lime-water also will deposit its calcareous earth upon the precipitate. After pouring off the water, and collecting the precipitate upon grey blotting paper, it will be found, that the calcareous earth thus precipitated by the perluted salt does not effervesce with acids, turns red litmus-paper blue, and disengages the volatile alkali from sal ammoniac on being rubbed in the hand with the same. But, if this precipitate is suffered to lie exposed to the air for only a day, all the above-mentioned properties disappear, and it now effervesces with acids. From this it appears, that the calcareous earth thus superfluously combined with perluted salt still retains the property of uniting with the aërial acid, which floats in our atmosphere; and hence

its effervescence with acids. Hence, too, we easily perceive what happened in Mr. Weber's precipitation of lime-water, viz. he employed too much lime-water; he dried the precipitate, triturated it, edulcorated it with water, and again exsiccated it. Here the superfluous calcareous earth had sufficient opportunity to imbibe fixed air from the atmosphere and water; hence his precipitate necessarily effervesced with acids; and as I did not add so much lime-water to the urine, my dried precipitate could not produce any effervescence. His experiment shewing that expressed oil renders *milk of lime* effervescent has not succeeded with me; but no doubt, this lime would effervesce with acids, if I, as Mr. Weber does, were to exsiccate it, then triturate it, and mix it with water. Who does not easily perceive, that, under such a tedious process in the open air, the lime absorbs aërial acid!

I have again distilled caustic spirit of sal ammoniac over a large quantity of fresh burned lime, saturated it with spirit of salt,

and obtained by sublimation a good sal ammoniac. Perhaps this spirit, too, was not perfectly caustic.

I assert, and Mr. Weber may safely believe me, that a fixed alkali may be rendered perfectly caustic by lime, and that such a caustic fixed alkali precipitates magnesia from acids, and precipitates it too, in a perfectly pure state. But Mr. Weber has not, perhaps, observed this phenomenon with sufficient attention; for the magnesian earth, when precipitated by caustic alkali, falls down almost transparent like boiled starch, or like the aluminous earth: Hence this precipitate easily eludes the sight: Whereas the precipitate which is obtained by means of the mild alkali, is much whiter, and much more visible.

Mr. Weber still brings forward an experiment, by which he endeavours to prove that calcareous earth dissolved in muriatic acid is capable of being mixed with Glauber's salt, without undergoing a complete decomposition. He saturates a pound of spirit of salt with calcareous earth, and adds

to this solution a full pound of Glauber's salt : After this salt is dissolved therein, he frees the mixture of its selenite, and separates the common salt from it by evaporation: When the solution becomes cold, he obtains Glauber's salt, and a good deal of mother-ley, consisting of calcareous earth and muriatic acid.

Struck with this experiment, and full of doubt respecting my own experience and that of all other chemists, I undertook to repeat it, in the same proportions as those assigned by Mr. Weber. I saturated a pound of common muriatic acid with calcareous earth, and tried to dissolve a pound of Glauber's salt in this solution ; but, notwithstanding the application of heat, a good deal of it remained undissolved. The mixture was as thick as honey ; so that I was obliged to add a quantity of water to it ; and the salt being now dissolved, I passed the solution through a filter, which became filled with selenite. Into one portion of the filtrated liquor I dropped some lixivium tartari, and obtained but very little precipitate, about as much as
might

might proceed from the selenite that was still held in solution. On evaporating this filtrated liquor, I obtained common salt, mixed with selenite; and after the ley was become cold, a quantity also of Glauber's salt. I evaporated the remainder further, and obtained the same salts, but not the smallest vestige or appearance of mother-ley. It is, therefore, impossible that Mr. Weber could have met with a quantity of mother-ley; and I cannot conceive how he conducted his experiments. I almost regret, I own, that I have bestowed so much time on this subject. I shall conclude in the same words with which Mr. Weber sums up his remarks: "The world may now judge who is most to blame."

IL. EXPERI.

XI. EXPERIMENTS

ON THE

ALKALINE SUBSTANCES

USED IN

BLEACHING;

By RICHARD KIRWAN Esq.

(CONTINUED.)

§ VI.

Of the best manner of procuring Alkaline Salts.

I. OF THE METHOD OF PROCURING MINERAL ALKALI.

MINERAL ALKALI may be procured more or less pure from the combustion of the various species of kali or *salsola* of Linnæus, or from that of the different species of *falicornia* and *chenopodia*, mentioned by the same author. The compounds thus formed are called *barilbas* or *soudes*.

The

The cultivation of the salicornia may be seen in the 5th vol. of the *Memoires des Scavants Etrangers*. A French acre (1,261 English) produces one ton of this weed ; and this ton when burned, produces but 100 weight of barilha, and this of a kind inferior to sweet barilha. A small quantity of this alkali is also contained in kelp. I am inclined to think that much of the alkali is lost by its union with the earthy parts during the fusion effected in the common manner of fabricating this substance ; and therefore the process suggested by Mr. Cadet may be useful. He advises a trench two feet deep, seven feet long, and eighteen inches broad, to be made, lined with clay mixed with sand, and over this iron bars two inches distant from each other to be laid ; upon which a wall 2,5 feet high is to be constructed, of lime-stone, if possible ; over the bars the dry sea-weed is to be laid and set fire to ; the ashes will fall into the trench, and when it is full the fire is discontinued*. I believe

* Mem. Paris, 1767.

also,

also, that washing the sea-weed in fresh water, to carry off the sea-salt that adheres to it, would be useful.

To desulphurate kelp, Abbé Mazeas recommends projecting on it while in a red heat, one-third of its weight of nitre; but this process seems too expensive to be practised in the great.

2dly. This alkali is found native in Egypt, and several parts of the Russian empire, and perhaps may be cheaply imported.

3dly. Common salt may be decomposed after Mr. Turner's method, by trituration with litharge, as the calx of lead here employed is afterwards converted into a yellow pigment. This method is very beneficial.

I have also contrived another process for decomposing common salt. The particulars of my experiment were as follows:

1st. I rendered the common salt pure by adding to its solution, a solution of mineral alkali until all the earthy matter was deposited.

2dly. To a solution of three ounces of this purified salt in nine ounces of water, I gradually

gradually added a saturate solution of 4.75 ounces of sugar of lead, both hot, until the solution of lead scarce excited any whiteness in that of the common salt. After one night's rest, part of the sugar of lead crystallized in the bottom of the vessel, by which it is plain that too much of it had been used. These crystals weighed 240 grains; the supernatant liquor I again evaporated to nearly two-thirds, and after two days obtained large pellicles of acetous soda, which I separated; they weighed 325 grains; to the residuum, which still had a sweetish taste, I added a solution of mineral alkali, until no further precipitation appeared; a very small quantity of the alkali was sufficient for this purpose. I then evaporated the remainder nearly to dryness; and afterwards heated it in a crucible to redness: In this heat it inflamed, and when calcined nearly to whiteness, I took it out and dissolved it in twelve ounces of water, filtered it, and on adding an hot solution of alum, obtained a precipitate, which, when dried, weighed 169 grains, and

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and indicated the quantity of pure alkali to be 112 grains nearly. In this process nothing is lost, for the lead may be either revived or turned into a pigment*.

* In the 5th number of Crell's Annals for 1789, the following method of separating the mineral alkali from common salt is recommended by Mr. Westrumb. Mr. Kirwan, no doubt, would have taken notice of it, had the account of it reached him before his own observations had been committed to the press.

1. Dissolve twenty pounds of common salt in sixty pounds of water, and to this solution add of pure dry potash 25 pounds, the larger lumps being previously pounded. This ley is to be evaporated till the saline pellicle which forms on the surface has sunk down several times, and been as often replaced by a new one. The kettle or boiler is then to be taken from the fire, and the liquor is to be suffered to cool a little, but not to become quite cold (between the temperature of 55 and 60 degrees of Fahrenheit) During the cooling, a large quantity of digestive salt will separate. The ley is now to be passed through a flannel strainer, that all the liquor may run off clear from the sediment. After this filtrated ley has stood about an hour—or until it is become quite cold—it will have deposited some more digestive salt, mixed with mineral alkali. The ley must, therefore, be poured off into another vessel, and be placed in a cold situation, where, if the directions have been properly followed, the mineral alkali will shoot into crystals, almost perfectly pure, and in greater abundance than is obtained by any of the common processes.

2. The saline matter which remains in the strainer consists of vitriolated tartar, and digestive salt. If the ley was suffered to become quite cold over these extraneous salts, it would, by reason of its oily consistence, be too much entangled in them; and the quantity remaining upon the filter is too considerable to admit of being purified like the rest from the mineral alkali. Hence, after pressing

Lastly, Glauber's salt may afford the mineral alkali, but most easily in the form of liver of sulphur: I endeavoured to decom-

pressing the saline mass together, let a little warm water be poured upon it, and let it afterwards be subjected, together with the salt that may have crystallized in the vessel after the first decantation, to the treatment hereafter mentioned. The digestive salt may be employed for making sal ammoniac, and for other purposes.

3. After the remaining ley has been poured off from the crystallized mineral alkali, let it be again evaporated, and be subjected, in other respects, to the same treatment which it underwent the first time, if much digestive salt should appear: If not, the ley may be immediately suffered to become quite cold, and the digestive salt which is collected, must be reserved for subsequent purification.

4. When the alkali has shot into crystals, the remaining ley is to be evaporated or inspissated, till all the salt is separated from it. Should any ley still remain, it may be set by for the next process; or it may be evaporated by a gentle heat till no more digestive salt is precipitated, and the alkali may then be suffered to crystallize in the cold.

The above-mentioned quantity of common salt, treated in this manner, generally yields 25 pounds of impure mineral alkali; and the process does not take up more than six or seven days.

5. The digestive salt obtained in the last process, and the alkali itself are purified in the following manner:

The first, viz. the digestive salt, is to be almost entirely dissolved by boiling in water, which is to be evaporated till about half of it is wasted, and the liquor is then to be placed in the cold. The digestive salt will first crystallize, and upon it the mineral alkali will afterwards be deposited in large crystals, which may very easily be separated. By repeating the evaporation a second time, the purification of the digestive salt will be fully effected. Should there remain any ley that still contains mineral alkali, it may be purified along with that which remains at last after the purification of the alkali.

6. The

pose it by the above process, but the quantity of alkali obtained from a large quantity of it was very inconsiderable.

OF THE VEGETABLE ALKALI.

It is universally known that this alkali may be extracted in greater or less quantity, by lixiviation, from the ashes of almost all ve-

6. The mineral alkali is purified by dissolving it in an equal quantity of water, and letting the solution stand some days in a cold place. If it contains a large admixture of digestive salt, this salt will appear on evaporating the liquor a second time (the first set of crystals of mineral alkali being previously separated from the liquor). At this time that portion of alkali which was obtained in the purification of the digestive salt (5) may be added. If the alkali happens to be pretty free from digestive salt, this salt will not shew itself till the third evaporation of the ley; by which time, if the operation has been conducted with a little skill and dexterity, almost all the alkali will have been separated.

By this method Mr. Westrumb generally obtained about twenty pounds of pure mineral alkali in large transparent crystals, and one pound and a half of alkali that was not so pure. The whole process does not take up, at farthest, more than twelve or fourteen days, and it may be finished in still less time by persons who are a little practised in such sort of operations, and who, without waiting to take each ley in exact order, seize opportunities of working upon different leys at the same time.

It is to be observed, that this method of separating the alkali from common salt succeeds only in the great.

Note by the editor of the Journal.

getables, and it is *now* well established that it pre-exists in vegetables before combustion; not indeed in a separate, uncombined state, but united partly with the vitriolic or marine acids, and sometimes the nitrous, but generally and for the greater part with a vegetable acid and oil, with which it forms essential salts, as they are termed. These vegetable acids and oils are decomposed during combustion, and thus the alkaline part is set free; but the vitriolic by contact with inflamed matter is converted into sulphur, part of which unites to the free alkali, which protects it from combustion, and thus forms what is called *liver of sulphur*, a product found in most ashes, especially when the air has not had free access to them during combustion.

As alkaline salts are of great importance in several arts, the proportion of ashes afforded by different vegetables, and that of alkali by the ashes of each sort of vegetable, has of late been accurately attended to. I shall here present the best authenticated results of the experiments made with this view.

One

One thousand weight of the following vegetables, perfectly dry and burned in a clean chimney and open fire, afforded the quantity of ashes and saline matter, exhibited in the annexed table :

One thousand lbs.	lbs. of ashes	lbs. of salt.
Stalks of Turkey } wheat or mais }	88,6	17,5
Of sun-flowers -	57,2	20
Vine branches -	34	5,5
Box - -	29	2,26
Sallow -	28	2,85
Elm - -	23,5	3,9
Oak -	13,5	1,5
Aspin - -	12,2	0,74
Beech -	5,8	1,27
Fir - -	3,4	0,45
Fern in August	36,46	4,25 <i>Home</i>
Wormwood -	97,44	73 <i>Wiegleb</i>
Fumitory -	219	79 <i>Idem.</i>

Table of the saline product of a thousand lbs. of ashes of the following vegetables :

	Saline Products.
Stalks of Turkey } wheat or mais }	- 198lbs.
K 2	Stalks

Saline Products.

Stalks of sun-flower	-	349	
Vine branches	-	162,6	
Elm	-	166	
Box	-	78	
Sallow	-	102	
Oak	-	111	
Aspin	-	61	
Beech	-	219	
Fir	-	132	
Fern cut in August	{ or	116, } <i>According</i> 125 } <i>to</i> <i>Wildenheim</i>	
Wormwood	-	748	
Fumitory	-	360	
Heath	-	115	<i>Wildenheim</i>

1st. Hence we see that in general weeds yield much more ashes, and their ashes much more salt than woods; and that, consequently, as to salts of the vegetable alkali kind, as potash, pearlash, cashup, &c. neither America, Trieste, nor the Northern countries, possess any advantage over us.

2dly. That of all weeds fumitory produces most salt, and next to it wormwood ;
but

but if we attend only to the quantity of salt in a given weight of ashes, the ashes of wormwood contain most. *Trifolium fibrinum* also produces more ashes and salt than fern.

Most of the experiments on woods were made in France by order of government, under the inspection of the overseers of the salt-petre works; yet are to be read with caution by those who attend to the quantity of alkali with respect to bleachers; for as tartar vitriolate (a salt useless to bleachers) is as serviceable to the makers of salt-petre as alkaline salts, they have constantly confounded one with the other; but the experiments made on weeds were instituted by persons who carefully discriminated these salts: 100 grains of the salt of wormwood contain but six of tartar vitriolate, and 100 grains of the salt of fumitory contain 15. All alkaline salts, unless mixed with lime, contain also one-fifth at least of fixed air, which produces no other effect in bleaching than that of restraining their activity.

§. VII.

*Of the process for obtaining POT and PEARL
ASH.*

1st. The weeds should be cut just before they seed, then spread, well dried, and gathered clean.

2dly. They should be burned within doors on a grate, and the ashes laid in a chest as fast as they are produced. If any charcoal be visible, it should be picked out and thrown back into the fire. If the weeds are moist much coal will be found. A close smothered fire, which has been recommended by some, is very prejudicial.

3dly. They should be lixiviated with twelve-times their weight of boiling water. A drop of the solution of sublimate corrosive will immediately discover when the water ceases to take up any more alkali. The earthy matter that remains is said to be a good manure for clayey soils.

4thly. The ley thus formed should be evaporated to dryness in iron pans; two or three at least of these should be used, and the
ley

ley as fast as it is concentrated passed from one to the other; thus much time is saved, as weak leys evaporate more quickly than the stronger. The salt thus procured is of a dark colour, and contains much extractive matter, and being formed in iron pots is called *pot ash*.

5thly. This salt should then be carried to a reverberatory furnace, in which the extractive matter is burned off and much of the water dissipated; hence it generally loses from ten to fifteen per cent. of its weight. Particular care should be taken that it should not melt, as the extractive matter would not be thoroughly consumed, and the alkali would form such an union with the earthy parts as could not easily be dissolved. I have added this caution, as Dr. Lewis and Mr. Dossie have inadvertently directed the contrary. This salt thus refined is called *pearl ash*, and must be the same as Dantzic pearl ash*.

* The French call the refined ash *potasse*, and the unrefined *salin*.
—Mr. Kirwan remarks that the alkali manufactured in the above-mentioned manner may not be sufficiently caustic for the earlier

For the most æconomical construction of a laboratory and furnaces for the above operations I refer to the description given in a French tract, called *L'Art de fabriquer le Salin et la Potasse*; and shall only add, that if the salt were extracted by a fire, supported by vegetables whose ashes might afterwards be employed, no inconsiderable advantage would be gained. Pearl ash is frequently tinged green or blue; this colour it acquires during fusion, not from any union of the salt with phlogiston, as was formerly supposed, but by reason of the manganese contained in the ashes of almost all vegetables, as Mr. Scheele has shewn. When it is calcined without melting, it is perfectly white as Dantzic pearl ash.

operations of bleaching; but by the addition of half a pound of quicklime to every 100 of the salt, or of ten pounds for every ton, it will be rendered sufficiently sharp. There is no danger that any of the lime will remain in the ley; but if any should, it will immediately be discovered and deposited by the addition of a little of the unmixed ley.

XII. EXPERI-

XII. EXPERIMENTS
ON THE
COLOURING MATTER OF LINEN
YARN, AND ITS SOLVENTS;

By RICHARD KIRWAN Esq.*

HAVING, through the obliging attention of Mr. Arbuthnot, procured a sufficient quantity of alkaline ley saturated with this colouring matter, or, as the workmen call it, *killed*, and which they are in the habit of throwing away; I found it to be a turbid liquor, of a reddish brown colour, a peculiar taste and strong smell, affording no sign either of acidity or alkalescence. On five quarts of this liquor I poured two ounces of weak marine acid; there was no effervescence, but a copious deposition instantly

* From the same vol. of Transactions as the preceding paper.

took

took place of a greyish green colour, and the liquor freed from this deposit was of the colour of red amber.

The next day I drew off the liquor with a siphon, and poured two quarts of pure water on the deposited matter; and having agitated the whole, suffered this matter again to subside, drew off the water, and added two quarts more; this liquor gave manifest signs of acidity, and continued somewhat reddish. Presuming that, after the addition of so much water, this acidity could not proceed from the small quantity of marine acid I had used, more especially as the liquor itself originally contained an alkali, in the saturation of which the greater part of the acid must have been employed, I began to suspect that this ley contained an acid of its own, which was disengaged and separated from the alkali by the marine acid as the more powerful of the two; and hence I reserved the two quarts of liquor, last added, for subsequent experiments.

After

After repeated affusions of cold water, when the characters of acidity were scarcely any longer perceptible, I threw the deposited matter on a filter, and suffered it to dry for some time; it was then of a dark greenish colour, somewhat clammy like moist clay. I took a small portion of it and added to it 60 times its weight of boiling water, but not a particle of it was dissolved. The remainder I dried in a sand heat; it then assumed a shining black colour, became more brittle, but internally remained of a greenish yellow, and weighed one ounce and a half.

By treating eight quarts more of the saturated ley in the same manner, I obtained a further quantity of the greenish deposit, on which I made the following experiments:

1st. Having digested a portion of it in rectified spirit of wine, it communicated to it a reddish hue, and was in great measure dissolved: but by the affusion of distilled water the solution became milky, and a white deposit was gradually formed; the black matter dissolved in the same manner.

2ndly.

2ndly. Neither the green nor the black matter was soluble in oil of turpentine or linseed oil by a long continued digestion.

3dly. The black matter being placed on a red hot iron, burned with a yellow flame and a black smoke, leaving a coaly residuum.

4thly. The green matter being put into the vitriolic, marine, and nitrous acids, communicated a brownish tinge to the two former, and a greenish to the latter, but did not seem in the least diminished.

Hence it appears that the matter extracted by alkalis from linen yarn is a peculiar sort of *resin*, different from pure resins only by its insolubility in essential oils, and in this respect resembling larks. I now proceeded to examine the power of the different alkalies on this substance. Eight grains of it being digested in a solution of crystallized mineral alkali saturated in the temperature of 60° , instantly communicated to the solution a dark brown colour; two measures (each of which would contain eleven penny weights of water) did not entirely dissolve this substance. Two measures

fures of the mild vegetable alkali dissolved the whole.

One measure of caustic mineral alkali, whose specific gravity was 1,053, dissolved nearly the whole, leaving only a white residuum.

One measure of caustic vegetable alkali, whose specific gravity was 1,039, dissolved the whole.

One measure of liver of sulphur, whose specific gravity was 1,170, dissolved the whole.

One measure of caustic volatile alkali dissolved also a portion of this matter.

Though these experiments were fully sufficient to resolve my doubts, yet to render still more satisfaction to bleachers, I repeated them with the salts they generally use, and also with soap.

I therefore dissolved one ounce of sweet barilha, Dantzic pearl ash, Cunnamara kelp, cashup and Clark's pearl ash, each in six ounces of pure water, and putting in one ounce measure of each solution eight grains of the green colouring matter, set them to digest

digest in a heat of about 180° for three hours and a half. At the end of this time, I found that the Dantzic dissolved more than the barilha; the kelp as much as the Dantzic; the cashup and Clarke's the whole.

Hence I added half an ounce more of the solutions of Dantzic, barilha and kelp; the Dantzic and kelp then dissolved the whole; but of the solution of barilha, two ounces were requisite to perform this effect.

I also dissolved half an ounce of Windsor soap in eighteen ounces of water; the solution was turbid, and could not be rendered transparent but when it was near boiling, and then it was very unmanageable; for when boiled it spouted three feet high out of the bottle. Three ounces of this solution were requisite to dissolve eight grains of the colouring matter.

Now to compare the powers of these different solvents we must remark, that as an ounce of barilha contains 115 grains of mere alkali, the solution of it being made in six ounces of water, each ounce of the

solution must contain the sixth part of 115, i. e. nineteen grains; and in the same manner it will be found that an ounce of the solution of Dantzic salt contains fifty grains of mere alkali, that of Cunnamara kelp 2,8 grains, cashup 15, Clarke's 21.

Therefore 4,2 grains of saline substance of kelp performed the same effect, as 75 of that of Dantzic, 38 of that of barilha, 15 of that of cashup, 21 of that of Clarke, 213 of soap.

I also tried the power of lime water, but found that three ounces of the strongest dissolved very little of the colouring matter, as should be naturally expected; for the three ounces did not contain above three grains of lime, nor did the mixture of sulphur render it more active.

From the foregoing experiments we may now deduce the following practical propositions:

1st. Liver of sulphur is of all alkaline compounds the strongest solvent of the colouring matter; next to this the caustic vegetable, and after this the caustic mineral alkali; the mild vegetable and mild

mild mineral alkali occupy the last place. Sulphur, it is said, leaves a stain in linen; but if liver of sulphur be used in the beginning, that is to say in bleaching the yarn, the stain will probably be removed by the purer alkalies afterwards used. Hence the solutions of kelp, cashup and markoft are advantageously used in the first processes of bleaching, for which Dantzic and sweet barilha are less fit; but six ton of kelp will be necessary to produce the same effect as one ton of cashup; yet as the former is manufactured at home it deserves the preference.

2ndly. As the alkali manufactured from inland weeds is more powerful than the mineral, Mr. Clarke's is more powerful, or may be rendered so, than any imported. It is already sufficiently caustic, and may be converted into liver of sulphur only by adding one-twentieth of its weight of sulphur to it when boiling, and thus it is fitted for the first processes of bleaching. In its primitive state it is fit for the second process, and by rendering it milder (which
may

may be effected by burning half a bushel of charcoal in a pan in the same room in which its solution stands) it will be adapted to the last processes, in which a less active alkali is required.

3dly. Clarke's salt converted into liver of sulphur is preferable to kelp, because this latter, by the present manner of manufacturing it, holds charcoal in solution; this coaly matter it deposits on the yarn, and thus leaves a black tinge; whereas Mr. Clarke's is free from this contamination, to say nothing of the far greater quantity of alkali it contains, insomuch that one ton of Mr. Clarke's is nearly equal to eight tons of kelp. Hence it clearly follows, that the linen manufacture stands in no sort of need of foreign salts or ashes for the processes of bleaching.

The chief defect in Mr. Clarke's manipulation is the loss of time during what he calls the *maceration* of ashes and quicklime; by barely moistening them the same effect may be produced in nine hours, which he expects from their maceration during

nine months, and much more lime is used than is necessary.

Dantzic pearl ash contains much more alkali than Clarke's; this must proceed from the superior quality of the ashes from which it is extracted. Those I received from Mr. Clarke were exceeding bad; nor do I believe that any crude ashes can be advantageously used in bleaching. But if some persons in the different manufacturing counties would allot a few acres to the culture of worm-wood and fumitory, I believe their own advantage, as well as that of the public, would thereby be considerably promoted. An acre will, I suppose, scarcely produce less than four tons of the dry weeds, and each ton will afford nearly 200 weight of ashes, and each ton of worm-wood ashes will give nearly 1500 weight of unrefined salt, or 1300 of the refined.

XIII. ON THE COMPONENT PARTS
OF THE
COLOURING MATTER OF PRUSSIAN BLUE;

By Mr. BINDHEIM.

(continued)

FROM the knowledge which we have already acquired of the blue-colouring substance, I concluded that as the presence of inflammable matter is not discoverable in the residuum of calx of iron, there certainly must be a second or third principle combined therewith. To discover the nature of this principle, one dram 44 grains of the basis of the colouring matter in Prussian-blue, obtained in the manner before-mentioned, were rubbed together with an equal quantity of caustic vegetable alkali, and the whole was then repeatedly steeped in hot distilled water, till the lixivium

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at length ceased to communicate a blue colour to a solution of iron. The filtrated lixivium was colourless, was easily affected by the solution of iron, and was exceedingly pure, as I was convinced from there being no precipitation on adding a solution of ponderous earth. It certainly did not contain any vitriolated neutral salt, with which the common Prussian lixivium is usually mixed; an imperfection which was first, and but lately, noticed by Messrs. Meyer and Klaproth, and which may be, and no doubt has been the cause of much chemical inaccuracy. Here, then, we have a new method of obtaining a remarkably good and exceedingly pure lixivium sanguinis, which, if it be not superior to the pure lixivium of this kind prepared according to the other known methods, does at least deserve to be placed on an equal footing with them. This lixivium was marked No. I.

The residuum was treated in like manner with fifty-six grains of caustic alkali and a sufficient quantity of water. I obtained

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tained again a phlogisticated lixivium, which, however, did not contain so much colouring matter as the first.— No. 2.

The residuum of this was again steeped with eighty grains of caustic alkali and water; this lixivium (No. 3) possessed very little of the colouring principle.

Lastly, that all the colouring matter might be extracted from the residuum, it was once more treated with two drams of caustic alkali and water as before. As none of the blue-colouring principle could be discovered in this lixivium (No. 4) I discontinued the treatment. There remained twenty-four grains of a black residuum, which, after being subjected to a red heat with inflammable matter proved to be phlogisticated iron. It had lost its blue-colouring property, and was no longer susceptible of inflammation on being held over a candle, upon a hot iron.

Thus the alkali was combined with the colouring substance, which I now wished to transfer to some other body. Accordingly,

I precipitated quicksilver from its solution in nitrous acid with Nos. 1, 2, and 3. In doing this, care must be taken that too much of the lixivium is not added to the solution; otherwise the mercurial precipitate will be redissolved. Of the precipitate thus obtained two drams and a half were well mixed with three times the quantity of charcoal dust, and put into a strong, well burnt, earthen retort, which was placed in a furnace, in which the heat could be pushed to the greatest intensity. To the neck of the retort was adapted a receiver half full of water, in the same manner as is practised in the preparation of phosphorus. In the degree of heat which was first applied, the quicksilver came over, and as the heat increased a phosphorescent vapour came steaming out of the retort into the receiver; and lastly, when the fire was urged still further, there appeared in the mouth of the retort a small quantity of shining phosphorus,—which, considering the small portion of precipitate that was used in this experiment, was perfectly

fectly satisfactory and decisive. Thus we are made acquainted with the hitherto unknown substance obtained in the decomposition of the colouring matter of Prussian-blue, and by this experiment it is proved too clearly to admit of any doubt, that the aforesaid substance is phosphoric acid.

As it may be doubted whether the before mentioned regulus of manganese in the humid way, properly deserves that name, I shall subjoin some observations in order to explain whence it happens, that, with the application of inflammable matter and proper treatment, it is still capable of taking up a portion of phlogiston, although possessed already of the properties of the regulus of manganese. After digesting some of it in nitrous acid, there remained on its outside a blackish matter, which it had before; the acid, however, had dissolved a part of it. On adding mild alkali to this solution, aërated calx of manganese was precipitated: With vitriolic acid the effect was the same. Hence we see that the regulus of manganese pre-

pared in the humid way so far agrees with that prepared in the dry way, that part of it is soluble, part not. However, as the quantity of the last, viz. of the insoluble part, was pretty considerable, owing to a want of, or imperfect saturation with, phlogiston; I endeavoured to remedy this defect by communicating to it the inflammable principle. I shall here relate in a few words the experiments which I made with this view, together with the phenomena which presented themselves.

Two drams and a half of the regulus of manganese prepared in the humid way, were mixed with one dram and a half of sugar, and, being put into a matrafs, two ounces of diluted nitrous acid were poured upon them. The acid was afterwards evaporated in a sand-bath. When the mass was become dry, and whilst it still remained in the heat, it gradually swelled up and enlarged in bulk, with an internal motion that proceeded regularly from the bottom upwards; till at length the mass took fire like phosphorus; and

and during its combustion a number of fine, thin flakes, of a dark brown colour, were thrown up and scattered partly against the sides of the matrafs and partly out of its mouth. This was a pleasing appearance, and no bad representation in miniature, of that grand and awefully majestic spectacle which nature exhibits in the instance of volcanic mountains. In this experiment, however, I was disappointed of my proper object; to accomplish this, therefore, I adopted a somewhat different process, founded on the property which the calx of manganese possesses of strongly attracting the inflammable principle in the humid way, and also upon this circumstance, that the aforesaid attraction is much increased by the presence of an acid.

Accordingly half an ounce of aërated calx of manganese and one scruple of charcoal powder were mixed together, and being put into a matrafs, as much nitrous acid was poured upon them as was necessary to dissolve the manganese. The whole was then
kept

kept over a fire in warm sand, till it became of a thick consistence, and nitrous fumes were discharged. It was then taken from the fire together with the sand-bath, the acid vapour still continuing to be extricated. In this manner a dry mass was obtained, which did not take fire; this accident, however, very readily happens, unless there be some caution and address in applying the proper degree of heat, and unless care be taken that the vessel do not remain too long upon the fire.

When all was cold, I found in the vessel a shining, metallic residuum, to which some of the nitrous acid and charcoal-dust adhered, so as to have the appearance of a scoria; but they were easily removed by several ablutions in water.

The regulus of manganese thus prepared in the humid way, was in its under part polished and shining; of an uneven surface, of a dusky white colour, and very brittle. By exposure to the air it becomes somewhat darker and less shining. With the assistance of heat, the mineral acids dissolved it, a small residuum excepted; and from these
solutions

solutions an aërated calx of manganese was precipitated by means of the mild alkali.

We are sorry that the smallness and closeness of the characters in which *****'s M.S. was written, together with blots that occurred in one or two places, where, as we since understand, no erasements were intended, should have given rise to several errata in the printing of the first part of Mr. Bindheim's dissertation, in the last No. of this Journal. In reperusing that paper, our readers will please to make the following corrections:

Page 47, l. 24, for "the natural manganese, which underwent through all its component parts a decomposition," read "the natural manganese; it underwent through all its component parts the same decomposition."

P. 50, l. 3, for "while in this experiment the red precipitate gave out vital air; however, this does not follow, nor can it follow," read "since the red precipitate used in this experiment is known to give out vital air. However, this did not happen, nor could it happen."

Ibid. l. 6, for "the vital air which was here found in the mixture to be considered as the parent of the fixed air in somewhat of a super-abundant quantity," read "the vital air, which pre-existed in the calx of mercury here employed, to be considered as the parent of the somewhat superabundant quantity of fixed air."

P. 52, l. 4, for "when a little of the aërated calx of manganese, mixed with water saturated with fixed air was set to freeze," read "when water saturated with fixed air, and mixed with a little of the aërated calx of manganese, was set to freeze."

Ibid. l. 13, for "dephlogisticated alkali," read "the phlogisticated alkali."

P. 53, l. 9, for "the colour itself," read "the colour thereof."

Ibid. l. 12, for "appeared," read "may be obtained."

P. 54, l. 4, for "with another in solution," read "with another if in solution."

P. 55,

P. 55, for "Perhaps this colouring substance is also present in many other, though with all the known neutral salts the case may not be the same as it is here with the vitriolated tartar," read "Perhaps this colouring substance also presents with many other, if not with all the known neutral salts, the same effects as in the case here with the vitriolated tartar."

Ibid. l. 15, for "From the experiments which follow it appears that the colouring matter of Prussian blue has a stronger attraction &c." read "From these experiments it follows, that the colouring matter of Prussian blue has, in the humid way, a stronger attraction to the aërated calx of manganese, than to the [mild] vegetable alkali."

XIV. EXPERIMENTS

WITH

DEPHLOGISTICATED MARINE ACID;

By Mr. SCHMEISSER.

(continued.)

I Performed a great number of experiments with the dephlogisticated marine acid; but as many of them coincided with those made by Mr. Scopoli and Hermbsstadt, and

exhibited the very same effects with various substances, it would be superfluous here to recapitulate them all. I shall, therefore, only relate such experiments as differed from their's.

When I suffered dephlogisticated marine acid air to pass through water, a wax candle burnt in it with a greater flame than before. The water through which the air had been transmitted, shewed, when paper stained with litmus was dipped in it, that it had absorbed a very slight portion of acid, like water impregnated with fixed air. However the air itself did not appear to me to be miscible with water in the same manner.

Phosphorus, fastened by means of a wire to the inner surface of a cork that exactly fitted the mouth of the bottle, instantly took fire, with an appearance which was so much the more beautiful as the air was now somewhat phlogisticated by several previous experiments with phlogistic bodies. The phosphorus projected its inflammable matter on all sides in the form of beautiful little stars

stars of a blue colour, which fell into the fluid that was at the bottom of the vessel, and that held some gold in solution. Hereupon, to my astonishment, a beautiful violet colour appeared, but from whence it proceeded I am still at a loss to explain. The stability of the colour makes it probable that it was owing to the gold. These appearances afforded great pleasure to me, and to those connoisseurs and amateurs of chemistry who were present when the experiments were made.

The air contained in the second bottle did not seem to be so much dephlogisticated. Paper stained with litmus, it is true, was deprived of its colour by it; but it had no effect upon a bit of leaf gold, with which I had armed a glass-rod: However, on letting fall upon it some drops of volatile spirit of sal ammoniac, it was immediately and wholly dissolved. As this circumstance struck me with some surprize, I endeavoured to ascertain what it was in the spirit of sal ammoniac that produced this effect. Accord-

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ingly, I repeated the experiment, taking another bottle filled with dephlogisticated marine acid air. Into this I introduced a glass rod armed with gold, and at the same time forced into the bottle some volatile alkaline air, whereupon I had the satisfaction to see the gold immediately dissolved, and the bottle filled with vapour. I shall examine this fact more thoroughly in order to ascertain how far the alkaline air contributed to render this weak marine acid air capable of dissolving the gold. The solution of gold collected from this and former experiments, I precipitated with volatile alkali; whereby I obtained about one grain of well edulcorated precipitate, which, on being laid upon a hot iron, discovered the property of a weak fulminating gold.

I have learned from different experiments, that common marine acid cannot be wholly converted into an ærial form by means of manganese. A proof of this we have in the great quantity of fluid which remained, after I had abstracted the dephlogisticated marine
acid

acid air, notwithstanding that the manganese was not yet completely phlogisticated. Although it be granted, therefore, that the acid which I employed contained a portion of water; yet this aqueous part could not amount to so much; for out of three ounces only thirty grains were lost, which give nearly the weight of the obtained dephlogisticated marine acid air.

To this fluid, which had the properties of a weak acid, nothing more is wanting, I imagine, for bringing it back again to the state of common marine acid, than a portion of specific heat combined with the marine acid basis. Although it is a very plausible theory, that of Mr. Kirwan's, which states that common marine acid is formed from a particular basis, phlogiston, and a determinate quantity of fixed air;—the dephlogisticated, on the other hand, from a combination of dephlogisticated marine acid basis with an excess of fixed air; yet my practical enquiries on this subject give me great reason to doubt the truth of this theory

theory, since I have never been able to discover that excess of fixed air: For, the dephlogisticated marine acid air never once rendered lime-water turbid, nor shewed the other properties of fixed air. This I shall notice more fully when I come to repeat my experiments, on which occasion I trust I shall have it in my power to bring forward several weighty objections to the theory respecting the origin and composition of the dephlogisticated marine acid air. In the mean while I cannot help mentioning one circumstance, which is, that among the number of the component parts of the common marine acid must be reckoned a portion of water, which is essential to it. I think I can shew that manganese does not produce marine acid in an aërial form directly in proportion to the quantity of vital air which it yields; but, that the portion of water which is always present in the acid, and is even taken up with it in an aërial form, constitutes a certain part of the obtained dephlogisticated marine acid air.

On mixing dephlogistified marine acid air (which had been already considerably phlogistified by different experiments) with water saturated with fixed air, I obtained an acid, which in every respect resembled common marine acid. This, at first, appears to be very much in favour of Mr. Kirwan's theory, and indeed to be quite reconcileable with it. But, if we accurately examine the substances here employed, viz. the fixed air, water, and phlogistified air (which still contained the proper basis) we shall find, that water (if not in a liquid form, at least in an aërial form, viz. as a part of the dephlogistified air) must be admitted as one of the component principles of the marine acid.

In the last place, I mixed two ounces of the weak sort of marine acid obtained after the dephlogistified marine acid air, with one ounce of pretty strong spirit of wine. During the mixture, an agreeable odour was emitted; but, the acid being absorbed by the spirit, this odour was soon lost. I took
two

two ounces more of the same spirit of wine, poured it into a glass-cylinder, containing eight cubic inches of dephlogisticated marine acid air. This air was presently absorbed by the spirit of wine, but possessed a strong aromatic smell. From this I abstracted one ounce, which had a remarkably fragrant odour, somewhat like that of nitrous æther, and was aromatic to the taste. On mixing it with some distilled water, I had the satisfaction to obtain about twenty grains of an æther (*naphtha*) which, when set on fire, burnt with a blue flame. As not only this experiment, but also several others of mine, differ from the excellent observations made by Dr. Hermbstadt; I shall publish an account of the same, as soon as I shall have convinced myself, by a further repetition of the experiments, of the certainty of my discoveries. In treating of the production of muriatic æther, I shall have recourse to my friend Kunsemuller's admirable theory respecting the production of æther.---The residuum from the dulci-

fied spirit of salt afforded a few grains of saccharine acid.

In the former part of Mr. Schmeisser's paper printed in the last number of this Journal, page 96, l. 21, for "These," read "This". P. 99, l. 12, for "fourish," read "roughish." Ibid. l. 20. for "coloured," read "discoloured." Page 100, l. 23, for "I now poured off," read "I had distilled off." P. 101, l. 1, for "It might however, be the effect of the strong fire upon the much used manganese," read "It might, however, be manganese itself forced over by means of the strong fire." Ibid. l. 4, for "four," read "rough."

The same circumstances with regard to *****'s hand-writing as those mentioned at page 155 betrayed the Editor into the above errata

XV. NEW EXPERIMENTS

WITH

CHARCOAL;

By Mr. LOWITZ.

§. I.

COMMON vinegar, on being boiled in a matrafs with charcoal-powder, became perfectly limpid like water.

§. II.

With this colourless vinegar I tried to prepare some terra foliata tartari (*alkali vegetabile acetatum*); but, although I took a great deal of pains and used a great deal of charcoal-powder, I never could render the salt white; for, during the exsiccation it constantly acquired a very dark brown, extractive appearance. For the production, therefore, of a dry, white terra foliata tartari, it is absolutely necessary to

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use

use vinegar that has been previously distilled.

§. III.

The following are some of the remarkable effects which take place in the purification of honey :

(1) As long as honey, diluted with a sufficient quantity of water, is boiled with charcoal-powder, a very peculiar and unpleasant smell is perceived.

(2) If the charcoal powder is not added to the honey and water (*Hydromel*) in a quantity sufficient for absorbing all the slimy or mucilaginous parts, the filtrated hydromel constantly appears of a semi-transparent, blackish colour; and this continues, till the necessary quantity of charcoal-powder is added : and then the liquor runs through the filter as clear as water.

(3) If the residuous charcoal-powder, which served to deprive the honey of its smell and slimy matter, be lixiviated with a large quantity of water ; the water will acquire

acquire a similar semi-pellucid black colour.

(4) If this black water be evaporated, the black matter will be deposited on the sides of the vessel in the form of a foot, that is very soft and unctuous to the touch.

That these effects are owing to the slimy parts of the honey, seems to be proved by the following experiments :

§. IV.

To a diluted solution of an ounce of gum arabic was gradually added charcoal-powder, by pounds; the mixture was well boiled, and a little of it was frequently filtered for examination.

The liquor, however, constantly ran through the blotting-paper turbid and dark coloured till 30 lbs. of charcoal powder, with a proportionate quantity of water for its dilution, had been mixed with it; and then the percolated liquor was clear. The whole of the filtrated liquor was now evaporated; but none of the gum was any

longer to be found in it, so that it must either have been decomposed or simply absorbed by the charcoal.

§. V.

Charcoal powder has the same effect upon other fluids, which contain either vegetable mucilage or animal gluten. They will not run clear through the filter, till they have been completely deprived of their mucilaginous or glutinous parts by the addition of a proper quantity of charcoal-powder.

§. VI.

Beer, milk and lemon-juice mixed with charcoal powder remain of a turbid black colour, until the latter is added in a quantity sufficient for depriving those fluids of all their mucilaginous, caseous, and oily parts; for which effect those fluids must be diluted with a prodigious quantity of water.

§. VII.

From these facts we may determine *à priori*, and without having recourse to experiments,

experiments, the cases in which this clarifying power of charcoal is not at all applicable; viz. it is not applicable to any of those substances, in whose mixtures and composition oily, gummy, mucous or gelatinous matter constitutes an essential and necessary part. On the other hand, the charcoal-powder may be advantageously employed in all those cases, in which we wish to separate and remove the above-mentioned principles.

§. VIII.

The charcoal-powder which remained after the purification of a mother-ley of phlogisticated alkali, communicated to the water with which it was lixiviated a beautiful, bright green colour.

§. IX.

Charcoal powder, over which a very empyreumatic distilled vinegar that had been concentrated by freezing, had been abstracted till the charcoal was become dry, displayed

played upon its surface all the colours of a peacock's tail.

§. X.

Another quantity of charcoal-powder, over which some common vinegar, that had been frozen, had been distilled with a strong heat till the charcoal became dry, in half an hour's time after mixture with water, was thrown into such a strong motion, resembling an effervescence, that, although the mixture filled the glass-jar in which it was made scarcely half way up, at first; yet it now ran over with a thick froth.

§. XI.

All sorts of glass-vessels, and other utensils, may be purified from long-retained smells of every kind, in the easiest and most perfect manner, by rinsing them out well with charcoal-powder, after their grosser impurities have been scoured off with sand and potash.

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§. XII.

In the common mode of clarifying honey a great deal of scum is separated. From this scum, we may obtain honey perfectly pure and clear, by diluting it with a proper quantity of water, and adding to it, while upon the fire, as much charcoal-powder as is necessary to make it filter off clear. The filtrated liquor is afterwards to be evaporated to a proper consistence.

§. XIII.

Upon the disagreeable bitter taste of salt-water, charcoal has not the least effect. This seems to me to prove, that its nauseous taste is not owing to bituminous matter, but to the earthy neutral salts; for the charcoal would certainly extract or absorb any bituminous matter from the water; whereas, upon salts the charcoal has no effect.

§. XIV.

Salt of hartshorn is rendered uncommonly white, on being well triturated with
an

an equal quantity of charcoal-powder, and put into a retort so as to fill it half way up. The remaining space within the retort is to be filled up with coarsely pounded charcoal, and the whole is then to be subjected to distillation.

§. XV.

A large quantity of acid of tartar (produced from thirty pounds of crystals of tartar) treated with charcoal-powder, was brought to the point of crystallization in the space of seventeen hours; and, notwithstanding the very strong and hasty boiling (during which, owing to the excessively hot sand, a black and burnt saline crust had attached itself to the inside of the matrafs) it was afterwards found to be perfectly limpid like water, and totally free from empyreuma.

§. XVI.

In the purification of common ardent spirits by means of charcoal, without the help of distillation, if too little charcoal-powder be added, the spirits will always retain

tain a blackish turbid appearance. But this black matter may be instantly and entirely separated from the spirits, by the addition of dry salt of tartar in such quantity as is sufficient for it to form with the water which it attracts from the spirits, a distinct fluid. As soon as the separation of the watery from the spirituous parts takes place, the black matter is seen floating upon the undermost fluid in the form of an extremely fine black pellicle. On the other hand, if to a pound of such turbid spirits, only a very little (a grain, for instance) of the alkali be added, the separation of the black sooty matter will not take place till after several days.

§. XVII.

I mixed three pounds of oil of hartshorn with two pounds of charcoal-powder, and put the mass, which had the consistence of a paste, into a cucurbit, throwing in afterwards two pounds more of charcoal-powder. After I had fixed a capital to the cucurbit, I placed it very deep in the sand, and proceeded to distillation. The animal oil came over uncommonly

monly slowly; and, notwithstanding the very great degree of heat that was applied, the distillation lasted thirty hours. The oil still continued to come over white and clear even towards the end of the operation, although the pot, together with the sand contained in it, was become quite red hot; and it was only at the very last part of the process that a little dark brown oil ascended

§. XVIII.

By a counter-experiment, in which I distilled three pounds of the same sort of oil of hartshorn by itself, I was fully convinced, that the addition of charcoal in this process, especially since it requires such a long continued and violent heat, is rather prejudicial than advantageous: For, when no charcoal was added, the operation was completely finished, with a gentle heat, in the course of twelve hours, and the obtained oil did not fall short of the former (§. 17) either in quantity or quality.

§. XIX.

A very extraordinary and useful property of charcoal is the power which it has of attracting

tracting and retaining, in the distillation by means of the water-bath, the strongest part of vinegar, and of not parting with the same, till it is subjected to the stronger heat of a sand-bath. In a former essay on the method which I discovered of concentrating vinegar so as to obtain it in a crystallized form, I have treated of this point very fully*. It is a circumstance no less remarkable, that the charcoal-powder at the very last part of the process, when urged by the strongest heat, yields scarcely any thing but mere water, though one should have supposed directly the contrary; for, in all other distillations of vinegar the strongest part of the acid comes over last.

§. XX.

After filling a glass funnel with charcoal coarsely pounded and riddled, and freed by ablution with water from its very fine dust, I poured upon it a few pounds of the black

* See Chemical Journal, Vol. I. p. 242. and Vol. II. p. 20.

concentrated

concentrated liquor which remains after the distillation of Hoffman's Anodyne Drops. The percolated liquor was every day poured back again upon the charcoal in the funnel; and thus in the course of two months, this black acid was rendered perfectly colourless.

§. XXI.

People whose breath smells strong from a scorbutic disposition of the gums, may at any time get perfectly rid of this bad smell, by rubbing and washing out the mouth and teeth thoroughly with fine charcoal-powder. I was led to this discovery by the effects of charcoal on putrid flesh*. By means of this very simple application the teeth are at the same time rendered beautifully white.

§. XXII.

Brown (or otherwise coloured) putrid, stinking water, was not only immediately deprived of its offensive smell by means of

* Crell Ann. 1788, Vol. II. p. 38.

charcoal-powder, but was also rendered transparent. Hence it would probably be of use for preserving fresh-water sweet during sea-voyages, to add about five pounds of coarse charcoal-powder to every cask of water, especially as the charcoal might easily be separated by filtrating the water, whenever wanted, through a linen bag.

§. XXIII.

I extracted the acid from ants by infusing them in boiling water. Part of this acid I mixed with charcoal-powder. The acid however was so much entangled in the fine charcoal-dust, that after passing through four folds of blotting-paper it was of a dark turbid appearance, and did not begin to become clear till six months had elapsed. The peculiar smell of the acid remained unaltered.

§. XXIV.

I saturated six pounds of the same acid of ants with purified pot ash, and expected that I should be able to separate all extra-

neous matter from the acid, merely by boiling it with charcoal-powder. But in this I was disappointed; for, notwithstanding I used four pounds of charcoal-powder, I could not make the ley filter off clear. It was constantly of a dark turbid appearance, and yet it ran through four folds of blotting-paper as readily as pure water does.

§. XXV.

I now perceived that the acid of ants, like vinegar (§. 2.) cannot be purified unless it be subjected to distillation. Now, as it is known that this acid is very apt to acquire an empyreuma during its distillation, I thought that in this case, at least, the charcoal might be employed with advantage. Accordingly, I put three pounds of this acid into a retort, added thereto one pound of charcoal-powder, and after wiping the inside of the neck of the retort as clean as possible, I proceeded to distillation in a sand-bath. During the distilla-

tion the materials swelled up and formed large vesicles or bubbles; and, what is the most surprising, the acid came over, even in the middle of the operation, tinged of a black colour by the fine particles of charcoal, notwithstanding that the rising and spirting up of the materials was wholly prevented by a cautious regulation of the heat. However, the fine charcoal-powder which came over with the acid, and which was in a manner volatilized, was afterwards easily separated by filtration. The acid itself was very weak, and that which came over towards the end of the distillation, notwithstanding the great quantity of charcoal-powder employed, was of a brown colour and empyreumatic. The residuum contained in the retort was strongly caked together, and imparted a very deep brown colour to water. From all these experiments with the acid of ants it appears, that charcoal produces a considerable effect upon it, but that it cannot be employed for the purification of the acid.

§. XXVI.

I let sixteen pounds of urine stand to putrefy for two months ; and afterwards mixed with it, while it was boiling, two pounds of charcoal-powder ; whereupon the bad smell immediately went off, and there only remained behind the strong odour of volatile alkali. In order to separate all the mucous and extractive parts, I evaporated it with some more charcoal-powder, to dryness. The dry residuum thus obtained, being lixiviated with water, afforded a liquor which was perfectly as clear as water, and which, after it was evaporated to the point of crystallization, had only a slight brown tinge, and remained fluid enough to allow the salts which it contained to shoot easily and regularly into beautiful, white crystals of cubical and other forms.

§. XXVII.

On pure alkali, saturated with the colouring matter of Prussian-blue, charcoal-powder has no other effect than that of depriving it of its yellow colour.

§. XXVII.

§. XXVIII.

A watery solution of liver of sulphur (prepared by fusion) was, by the addition of charcoal without the assistance of heat, rendered perfectly colourless like water, and inodorous; but it tasted bitter like vitriolated tartar. On adding oil of vitriol to it, no precipitation ensued, nor was there any smell of hepatic air. However, both these effects took place, though in a very weak degree, after some hours had elapsed; for the previously colourless ley gradually acquired a yellowish colour, and became somewhat turbid. Five drams and a half of the solution of liver of sulphur deprived of its colour by means of charcoal, and an equal quantity of a solution of liver of sulphur that had not been treated with charcoal, were evaporated to dryness, each by itself, in a glass vessel. The former yielded 18 grains of a perfectly white saline residuum, which being laid upon burning charcoal entered into a red fusion, without shewing any mark of sulphur; the latter,

on the other hand, gave 22 grains of a blackish saline residuum, which, on being laid upon heated charcoal, broke out into a blue flame, emitting at the same time a strong sulphureous smell. Hence it appears, that sulphur dissolved by the fixed alkali, is deprived of its principle of inflammability by charcoal.

§. XXIX.

If a small quantity of a balsam of sulphur prepared with some essential oil, is dissolved in highly rectified spirit of wine, and charcoal powder is afterwards mixed therewith; the mixture is entirely deprived of the peculiar, unpleasant smell of balsam of sulphur, and there only remains the smell of the essential oil employed to dissolve the sulphur.

§. XXX.

Neither the watery nor the spirituous solution of soap seem to undergo any change by treatment with charcoal.

(The continuation of this subject in the next Number.)

XVI. EXPERI.

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XVI. EXPERIMENTS

ON THE

**PURIFICATION OF CRUDE NITRE,
OR, SALT-PETRE,**

BY MEANS OF

CHARCOAL - POWDER;

By PROFESSOR GADOLIN*.

§. I.

CRUDE NITRE or salt-petre contains various impurities, of which the most considerable are common salt, and digestive salt; magnesian and calcareous earths combined with marine or nitrous acid; together with an unctuous matter that is often ac-

* Extracted from a treatise of Mr. Gadolin's published in the Trans. of the Acad. of Sciences at Stockholm, and sent by the author himself to Dr. Crell.

accompanied with volatile alkali. The difference which takes place in the crystallization between the first mentioned salts and nitre, and the deliquescent property of the earthy neutral salts, afford sufficient means of separating each kind of salt from the other. If there happens, however, to be a great deal of the unctuous matter in the mixture, then neither the crystallization nor the separation of the heterogeneous salts can go on properly.

§. II.

The purification of crude nitre, therefore, depends principally on the separation of this fat; a separation, which (in the great way) is in part effected, by subjecting a strong solution of the nitre to boil, during which the unctuous matter separates in the form of a scum. However, a considerable quantity of it, that is more intimately combined with the salts, still remains with the nitre, and adulterates it so as to render it unfit for very nice purposes, till it has been further purified.

§. III.

§. III.

The best method hitherto proposed of freeing the nitre from this unctuous matter, seems to have been the addition of alum. But even if no objection lay against this method on account of its being too expensive to be adopted in the great operations, still there would be an objection from the apprehension that the nitre might take up a portion of the vitriolic acid, especially if the crude nitre should contain no great quantity of calcareous earth. It was therefore to be wished, that some other method might be found out of freeing nitre from all its impurities, without being attended with great expence, and suited to operations in the great.

§. IV.

As it has been lately discovered that different substances, whether saline or spirituous, charged with unctuous impurities, can be very well freed from the same, if boiled or
digested

digested with charcoal-powder*; it occurred to me that crude nitre might be purified most

* It was long since known that well burnt charcoal absorbed a good deal from the air and other fluids with which it happened to be surrounded; but new experiments were wanting to discover the property which it possesses of purifying fluid bodies from their viscous, oily or slimy parts; which impurities, when once absorbed by the charcoal, are retained in its interstices by means of their thick and glutinous quality. In my country some of our clever housewives have long boasted of being able to purify empyreumatic ardent spirits by putting into the still some birch-charcoal; and also that they could obtain a fine and clarified beer, by scraping down some soot from the chimney into the wort while it was boiling. While some have given credit to and adopted these housewifery arcana, others have disbelieved and despised them; and hence they have remained generally unknown, for want of proper experiments to establish the facts, and for want of a proper explanation of what takes place in the process.—It was Mr. Lowitz of Petersburg, who, as far as I know, first clearly pointed out how acid of tartar, ardent spirits, and many other substances may be freed from their unctuous impurities by means of charcoal. He imagined that the charcoal has a strong attraction for phlogiston, and upon this principle he concluded that it would dephlogisticate bodies in the humid way. In this manner he accounts for his method of purification (Crell, Ann. 1786, 1788). The experiments of Mr. Lowitz were repeated and confirmed by Mr. Piepenbring (1789) and by Mr. Brugnatelli and Westrumb (1789). On the other hand, Mr. Fuchs (1788) Hahnemann (1789) and others, who failed in their attempts to deprive saline solutions of their brown colour and tenacity, by means of charcoal, have disputed the purifying property assigned to charcoal by Mr. Lowitz. It is to be observed, however, that in many of these experiments an extremely pure charcoal is required; and hence the want of success in their

perfectly and easily in the same manner : Accordingly, I undertook, with this view, the following experiments.

§. V.

The crude nitre, of which I had procured twelve pounds, was very brown and moist. At the bottom of the vessel in which it was put, a dark brown fluid had collected, amounting to about five ounces and a half, which I separated, and shall call the motherley.

(a) Four ounces of the nitre spread out upon bibulous paper, and dried in a moderate temperature of the air, weighed 7,15 half-ounces.

their attempts was probably owing to the charcoal being badly burnt, or afterwards rendered impure by moisture which it had absorbed before it was employed.—Having repeated some of Mr. L.'s experiments and found them to be accurate, I was led to hope that salt-petre might be purified by means of charcoal-powder, which to my knowledge no person had ever tried before. This appeared to me to be an object of so much the more importance, as in the preparation of gunpowder so much depends on the purity of the nitre.

(b) Half

(b) Half an ounce of crude nitre dissolved in water gave, with a solution of silver, 0,0496 half ounce of dry luna cornea.

(c) Two ounces and a half of the crude nitre, dissolved in water, and supersaturated with vegetable alkali, gave a small quantity of a dark brown precipitate, which was not increased by a strong digestion (during which however, a smell of volatile alkali was perceived). The weight of the precipitate was 0,013 half-ounce.

(d) Half an ounce of the mother-ley gave with a solution of silver 0,368 half ounce of luna cornea. The ley being filtered and evaporated, yielded a quantity of transparent, prismatic crystals, some of which deliquesced in the air, but others shewed themselves to be pure nitre.

(e) From one ounce of the mother-ley, precipitated by means of the vegetable alkali, I obtained 0,092 half ounce of a triple coloured powder. The marine acid dissolved this powder with violent effervescence. The solution was of a dark colour and semi-transparent

transparent ; by filtration it was rendered clear, a fat scum being left upon the paper. Vitriolic acid dropped into it, rendered it turbid ; but on adding to it a sufficient quantity of water, it became clear again.— The vitriolic acid effervesced in like manner with the precipitate, and left a considerable quantity of undissolved gypsum in a powdery state. This solution when evaporated, afforded selenitic needles, and crystals of Epsom salt.

(f) One ounce of the mother-ley, evaporated to dryness by a gentle heat, left behind a dark brown mass, which was full of irregular cubic crystals, and which weighed 0,625 half ounce. In a cold room it attracted moisture ; dissolved in water and filtered, it left scarcely any thing on the filter. On adding to it a solution of mercury in nitrous acid, it threw down a copious white precipitate, which, however, was for the most part redissolved by the water. The light grey powder, which remained behind on the filter, discovered with the blow-pipe a thick vapour

vapour of caustic sublimate, and was converted at last into a black coal, which was gradually consumed by the flame of the blow-pipe till at last nothing remained but a very small quantity of ashes.

(g). Three ounces of the mother-ley, mixed with four ounces of water, and 3-4ths of an ounce of charcoal-powder*, were boiled together smartly for ten minutes; after which the whole was poured boiling hot into a filter, and lixiviated with six ounces of water of the same heat; what went through the filter was clear, and of a light yellow colour: It was again boiled up, with 3-4ths of an ounce of charcoal powder, then filtered and lixiviated as before. It now passed much quicker through the filter, and was nearly as clear as water. The ley, which now amounted to fifteen ounces, being re-

* Not only in this but in the following experiments I have employed well burnt charcoal prepared from deal or the wood of the fir, such as was quite free from ashes. It is not to be doubted but the same effects will follow, if charcoal of other wood be employed, provided all its oily particles be thoroughly diffipated by fire.

duced

duced by evaporation to six ounces, and afterwards set by in a cold place; there first shot forth in it several white cubic crystals of digestive salt, and then some fine prismatic crystals of nitre. These crystallized salts weighed altogether, 0,9 half-ounce. After evaporating the lixivium still further, I obtained 0,17 half ounce of cubic crystals, which evidently smelled of marine acid, when dried with the assistance of heat; and left a small quantity of a white powder undissolved, when lixivated with water. The solution of these crystallized salts gave, with the addition of vegetable alkali, 0,06 half-ounce of a light gray powder; and the last lixivium which could not be made to crystallize yielded, on the addition of an alkali, 0,116 half-ounce of a precipitate which consisted of calcareous and magnesian earths. The charcoal had acquired an increase of $\frac{1}{4}$ th of an ounce in its weight.

§. VI.

From the foregoing experiments it follows, that 100 parts of the crude salt-petre
which

which I employed, contained nearly $9\frac{1}{2}$ per cent. of mother-ley (a) and $3\text{-}7$ ths more of marine acid than the same quantity of mother-ley. Again, according to experiment (b) 100 parts of crude nitre contain as much marine acid as 4,96 parts of luna cornea; and according to experiment (d) in $9\frac{1}{2}$ parts of mother-ley there was only as much marine acid as in 3-5 parts of luna cornea. This proportion, however, may not be perfectly accurate; for the unctuous matter of the solution being seized by the metallic calx, the weight of the precipitate would be thereby increased, as may be clearly seen from the experiment (f) with quicksilver. But for this trifling impurity, so much the less allowance can be made in the case of the silver-precipitate, as I observed, on the other hand, that my luna cornea was unusually soluble in water, and consequently during the lixiviation was diminished in quantity, more than a pure luna cornea generally is. This was probably owing to the presence of volatile alkali in the mother-
ley

ley, which renders muriated silver soluble in water.

§. VII.

From experiment (g) we learn that 100 parts of the mother-ley contain 8,5 parts of the unctuous matter; and as the whole of what was dissolved in this amounted to 31 per cent. (according to experiment f) it follows that the salts contained in the mother-ley, which for the most part consisted of marine acid, vegetable alkali, calcareous earth, and magnesia, gave 22,5 per cent. --- Consequently the ingredients in 100 parts of unrefined salt-petre were about 89 parts of pure nitre, three parts of muriated neutral salts, one part of unctuous matter, and six parts and a half of water, without reckoning in the water of crystallization.

§. VIII.

As I perceived from experiment (g) that even the mother-ley itself could be freed from its unctuous matter by means of charcoal; I had no longer any doubts about the pos-

sibility of purifying crude nitre by the same means. It only remained for me to ascertain what proportion of charcoal-powder the salt petre requires, and what phenomena present themselves in the process—The four following experiments were conducted in one and the same manner, viz. in each I took one pound of unrefined salt-petre, and boiled it briskly with charcoal and six pounds of water, for ten minutes, in a copper vessel, and afterwards filtered it, while it was boiling hot, through doubled blotting paper. The filter was then lixiviated with two pounds of boiling water; and the filtrated lixivium was evaporated, till a drop of it, let fall upon cold glass, instantly began to crystallize. The ley was then poured into a wide glass vessel, and set by in a cold place to crystallize. In this manner I obtained from the first crystallization about ten ounces of crystals of nitre. The ley which was then poured off, being further evaporated, afforded three ounces and a quarter, and in the third crystallization, one ounce

ounce more of nitre. The last lixivium still yielded a little nitre, with a considerable quantity of digestive salt.

First experiment. When one pound of crude nitre was boiled (in the above-mentioned manner) with four ounces of charcoal-powder, a lixivium as clear as water was obtained, and the crystals of the first and second formation were perfectly white and transparent. Of both these sets of crystals a quarter of an ounce (each separately) was dissolved in one ounce and three quarters of water, which, on the addition of some solution of silver, became milky, and deposited (each solution) a precipitate that weighed 0,002 half ounce.--The crystals of the third formation were still very transparent, and remarkably whiter than those of the common purified nitre. From a quarter of an ounce of these I obtained, by means of the silver-solution, 0,007 half ounce of luna cornea.---The crystals of the fourth formation, which contained a greater admixture of calcareous salt, were still very white, but not quite so transparent as the former.—The last mother-

ley was become somewhat of a yellowish colour, yet it gave with the vegetable fixed alkali a perfectly white precipitate, which weighed $\frac{1}{10}$ half ounce, and consisted of calcareous and magnesian earths.

Second experiment. One pound of crude nitre, treated as above with two ounces of charcoal-powder, gave a clear, but somewhat yellowish lixivium. The crystals of the first, second, and third formation did not differ at all in their external appearance from those obtained in the first experiment. The results too, with the solution of silver were very similar to those in the former case; thus, a quarter of an ounce of the two first sets of crystals gave 0,002 half ounce of luna cornea, and one quarter of an ounce of the last set or formation gave 0,008 of luna cornea. The fourth set of crystals had a yellowish tinge, and the last mother-ley was of an orange colour.

Third experiment. From one pound of unrefined salt-petre and one ounce of charcoal-powder I obtained a yellowish lixivium,
and

and in the first crystallization, very white and transparent crystals, with a yellowish tinge too slight to be observed, except by closely comparing them with the crystals obtained in the two former experiments.--- A quarter of an ounce of these crystals gave with the solution of silver 0,0006 half ounce of luna cornea.---The second set of crystals were very similar to the former, but with the silver-solution they afforded 0,0026 half ounce of luna cornea.---Those of the third crystallization had a much yellower appearance, like nitre refined in the common way; a quarter of an ounce of them precipitated from the solution of silver 0,012 half ounce of luna cornea.---The crystals of the fourth formation were of a straw-colour. The last lixivium was of a dusky sorrel colour.

Fourth experiment. One pound of crude nitre treated with half an ounce of charcoal-powder, gave a dark orange-coloured lixivium, the first set of crystals from which were tolerably white, but not so transparent

as those of the preceding experiment. A quarter of an ounce of them, dissolved in one ounce and three quarters of water, were rendered milky by the addition of the silver-solution, but no sediment was deposited. The filtrated liquor was clear, though the paper through which it had passed gained no increase of weight, or at the utmost not more than 0,0001 half ounce. The second set of crystals resembled those that were obtained in the third crystallization. From a quarter of an ounce of them I had 0,0034 half ounce of luna cornea. The crystals of the third formation were yellowish, and a quarter of an ounce of them gave, with the solution of silver, 0,0135 half ounce of luna cornea. The last lixivium was of a dark brown colour.

Fifth experiment. One pound of crude nitre was dissolved in six pounds of water, and boiled, as above, but without the addition of charcoal-powder. The filtrated lixivium was of a dark orange-colour. The nitre of the first crystallization was somewhat

what yellowish, like that of the third crystallization in the first experiment. A quarter of an ounce of it afforded 0,001 half ounce of luna cornea. That of the second crystallization was yellower, and, with the solution of silver, gave 0,0045 half ounce of precipitate. A quarter of an ounce of the yellow crystals of the third formation yielded 0,017 half-ounce of precipitate with the silver-solution. The last lixivium was of a very dark brown colour.

§. IX.

It is remarkable that the nitre obtained in the first crystallization, from ley that was most free from unctuous matter, gave more luna cornea with the solution of silver, than the nitre that was obtained from the less purified ley. This circumstance (if the unctuous matter does not, which I think is very probable, render some of the luna cornea soluble) might be owing to the too great disposition which the highly purified nitre has to crystallize, for under such a quick

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crystalliza-

crystallization the muriated salts have not time to detach themselves. On the other hand, if the ley be too unctuous and tenacious, a greater quantity of muriatic salt will be retained in the nitre, and the crystallization will not take place in a proper manner. This inconvenience from pushing the purification too far, is not likely to happen when large quantities are operated upon; for in that case the cooling goes on very slowly, and the crystallization always proceeds in a much more regular manner. And after all, this greater retention of common salt (which only happens when the whole quantity of common salt contained in the ley is very inconsiderable) does not exceed $\frac{1}{2}$ per cent. which small quantity is of far less consequence in the making of gunpowder, than even the slightest portion of unctuous matter.—On the other hand, the advantage of having the lixivium thoroughly free from its unctuous matter is obvious, even with respect to the admixture of common salt, if the quantity contained

tained in the ley be considerable. Thus, the leys in the second crystallization contained ten or twelve per cent. (relatively to the nitre) of common salt; but the crystals, where the unctuous matter had been removed, yielded only as much marine acid as is contained in $\frac{1}{3}$ per cent. of common salt: Whereas, where little or no charcoal-powder was employed in the purification, the admixture of common salt amounted to between one quarter and $\frac{3}{8}$ per cent. And, in the third crystallization, where the whole admixture of common salt amounted to thirty per cent, the nitre which had been purified by means of charcoal was found to contain only $\frac{2}{3}$ per cent. of common salt, whilst that which had been purified without charcoal-powder contained above twice as much, or one and a half per cent, of common salt.

§. X.

Concluding from the preceding observations, that $\frac{3}{4}$ of an ounce of charcoal-powder

powder would be sufficient for separating from the ley as much unctuous matter, as would render the crystals of the first formation perfectly free from unctuosify; I subjected the remainder of my crude nitre to the following experiments. One pound of unrefined salt-petre was boiled for ten minutes with $\frac{3}{4}$ of an ounce of charcoal-powder and six pounds of water; the liquor was then filtered, and the charcoal-powder lixiviated with two pounds of boiling water. The percolated liquor was evaporated to the point of crystallization, and after the crystallization had taken place, the clear lixivium was poured off, and being mixed with twelve ounces of crude nitre and three quarters of an ounce of charcoal-powder and a proper quantity of water, it was again boiled, filtered, and crystallized. This, as is seen in the following table, I repeated several times, and thus obtained in each crystallization very white and transparent crystals of nitre.

The

The first column of the table shews the weight of the crude salt-petre employed. The second, the charcoal mixed with it. The third, the crystals of nitre which formed. Fourth, the weight of the silver-precipitate, obtained from a quarter of an ounce of the crystals. Fifth, the precipitate of silver from the same crystals, reckoned in the ratio of per cent. to the salt-petre. Sixth, the precipitate of silver from the crude nitre of the leys, reckoning from §. V. (b) and in the ratio of per cent. to the pure nitre.

TABLE

TABLE.

I.	II.	III.	IV.	V.	VI.
Crude Nitre.	Charcoal-pow- der.	Crystallized Nitre.	Luna Cornea, from a quarter of an ounce of the crystalliz- ed nitre.	Luna Cornea, from 100 parts of crystallized nitre.	Luna Cornea from the livi- um, to 100 parts of pure nitre.
Half Ounces.	Half Ounce.	Half Ounce.	Half Ounce.	Parts.	Parts.
32	2,5	19,5	0,0009	0,18	5,5
24	2,5	21,	0,0011	0,22	3,
24	2,5	20,9	0,0013	0,26	13,
24	2,5	20,4	0,0017	0,34	16,
24	2,5	22,25	0,0025	0,50	20,
24	2,5	20,	0,0018	0,36	24,
24	2,5	21,5	0,0020	0,40	27,
0	1,	6,75	0,0070	1,4	90,
0	0,	2,5	0,179	35,8	300,
1					
2					
3					
4					
5					
6					
7					
8					
9					

Before the last crystals (No. 9) had shot forth, a thick saline crust had formed upon the surface of the ley; and during the cooling there shot forth a great number of cubic crystals, from which I first decanted the lixivium, and afterwards obtained the three fourths of an ounce of crystals marked in the table.—The crust, together with the cubic crystals, being dissolved in a little water, gave an insoluble residuum of white selenite in a powdery form, amounting to 6,06 half ounce. But the solution, as well as the last lixivium, gave with vegetable alkali an earthy precipitate weighing $\frac{3}{4}$ half ounce, and consisting of calcareous and magnesian earths, together with a little unctuous matter.

§. XI.

From the preceding table we see how little disposed the muriated salts are to unite with the nitre, when the lixivium is freed from its unctuous matter, and the crystallization is properly conducted. But
I shall

I shall not dwell upon this point, my only design being to shew, how admirably well charcoal-powder answers for depriving nitre of its unctuousity, and that, by means of it, not only a perfectly pure nitre may be obtained in the first crystallization, but also that the lixivium separated from the crystals proves an equally useful addition in the subsequent purifications, and crystallizations. And since (according to §. V. g.) the mother-ley itself is rendered fit for crystallization by means of charcoal-powder, there is reason to believe that charcoal-powder might be very advantageously employed by the salt-petre makers in the first boilings of the impure ley.

§. XII.

As all operations in the great require, in many respects, a different treatment from those which are performed in the small way; so, from the above-related experiments I can guess with some degree of certainty what should be the proportion of charcoal-powder

powder to the nitre in large purifications. In all probability much less than $\frac{3}{4}$ of an ounce to the pound will be sufficient. This and other circumstances will be learned from practice.

If, in large purifications, the unctuous matter should be converted into scum, the charcoal-powder will probably be separated from the ley by the despumation; in which case filtration will be unnecessary, and the expence attending this new process will then be very trifling. But even though the filtration should be found necessary, still I am persuaded that the expence, whatever it be, will be amply compensated by the greater purity of the nitre, and by the superior quality of the gunpowder prepared from it.

XVII. CHEMICAL NEWS.

SINCE his analysis of the seminal liquor*, Mr. Vauquelin, in conjunction with Mr. Fourcroy, has proceeded to examine (*Annales de Chimie* for August, 1791) the lachrymal and nasal humours. During the spontaneous evaporation of the former (viz. tears) in a dry air, some cubical crystals shot forth in the midst of a mucilage, which (to use the author's expression) served as a mother-water. These crystals being separated therefrom by means of spirit of wine, which dissolves them without attacking the mucous matter, shewed the same properties as common salt; with this difference, however, that a solution of them turned paper stained with blue vegetable colours, green, in consequence (as was afterwards discovered) of the presence of a little loose mineral alkali.—Dephlogisticated marine acid coagulates the lachrymal fluid into white flakes,

* See the First Volume of this Journal, page 307.

which

which become yellow if the acid is added in sufficient quantity. These flakes are insoluble in water. After the action of the vitriolic acid upon the exsiccated lachrymal humour, nothing remains but some Glauber's salt mixed with the mucous matter. After the action of the marine acid, the only salt contained in the mucilage is common salt. These two experiments prove that tears contain common salt and mineral alkali. This last is supposed to exist in the lachrymal humour in a pure or caustic state, because lime-water occasions no precipitation when mixed with fresh tears, but is rendered turbid by the product which is obtained after the spontaneous evaporation of the lachrymal fluid in a dry air.—Spirit of wine added in sufficient quantity, decomposes and precipitates the lachrymal humour under the form of large white flakes. If the spirit of wine, after it has been separated from the precipitated mucus, is evaporated, it leaves behind a little common salt and mineral alkali.—The product ob-

tained from the lachrymal humour inspissated by spontaneous evaporation in the air, being burnt to ashes gave marks of the presence of phosphorated calcareous earth, and also of phosphorated mineral alkali, which, however, is but barely perceivable. As for phosphorated volatile alkali (phosphoric ammoniac) it is evident there could not be any of that, on account of the presence of loose mineral alkali, by which it would necessarily be decomposed. From these experiments it follows, that the lachrymal humour consists of water (which constitutes the greatest part of it) a particular mucilage, common salt, mineral alkali, and lastly, phosphorated calcareous earth and phosphorated mineral alkali; the proportion of these last, however, is so extremely small, as to be but barely perceptible.

The nasal humour yielded nearly the same results.

Mr. Patrin (*ibid.*) having procured from the mines of Zmeof, in Siberia, a fossil, which in its external characters resembled

the terra ponderosa aerata of Alston Moor, first analysed by Dr. Withering, and afterwards by Messrs. Sage and Fourcroy, engaged Mr. Pelletier to undertake the analysis of it; which he accordingly did, and proved from his experiments that it was a true aërated barytes. Mr. Pelletier thinks that this fossil occurs more frequently than is supposed, and that it is probably mistaken amongst miners for the marmor metallicum or ponderous spar.

Mr. Fourcroy (*Annales de Chimie* for September 1791) has obtained a new triple salt, by combining the volatile alkali with vitriol of quicksilver. When *a little* spirit of sal ammoniac is added to a solution of a pure and neutral vitriol of quicksilver, a copious grey precipitate is thrown down, which, on being exposed upon its filter to the rays of the sun, is in part reduced to running mercury; another portion of this precipitate remains in the form of a dark grey powder, without being revived. This last is completely redissoluble in spirit of

sal ammoniac, and is the triple salt above-mentioned, viz. *Ammoniated vitriol of quicksilver* (*sulphate ammoniaco-mercuriel*): On the other hand, if a large quantity of the spirit of sal ammoniac is added to the solution, the precipitate is much less abundant, but is of a much darker colour, and is completely revived by the contact of light, and more especially by exposure to the rays of the sun. The supernatant liquor contains the triple salt, the ammoniated vitriol of quicksilver, in greater abundance than in the preceding case.—If, instead of decomposing the neutral vitriol of quicksilver dissolved in water, spirit of sal ammoniac is poured upon the salt in its solid form, and in a thoroughly dry state, there arises a strong effervescence, accompanied with a noise like that which takes place on plunging a red hot iron into water; this effect is owing to the extrication of phlogisticated air. There is at the same time deposited at the bottom of the ammoniacal spirit, a very black powder, which is completely and quickly reduced to running mercury by the contact of light. The supernatant

pernatant liquor contains the triple salt, the ammoniated vitriol of quicksilver.---- To obtain this salt in a regular and crystallized form, its solution must be evaporated, either spontaneously in the air or with the assistance of a gentle heat. In this way (provided the vitriol of quicksilver has been precipitated by a sufficiently large quantity of spirit of sal ammoniac) very shining, firm, polygonal crystals are obtained.—If instead of letting the solution deposit crystals spontaneously by exposure to the air, a large quantity of distilled water is added to the solution, it immediately becomes white, milky and opaque, throwing down a white precipitate, which is a triple salt, like the before-mentioned crystallized salt, but which does not appear in a crystallized form, because its separation takes place too suddenly.—The liquor which remains after the precipitation of the solution by the distilled water, yields by evaporation a shining lamellated salt, which has a pungent austere taste, and which consists

consists almost entirely of vitriolic ammoniac.---The triple salt, whether obtained by crystallization or by diluting the solution largely with water, is in fact (though there appears to be a difference) of one and the same nature; except that the former contains an excess of volatile alkali, the latter an excess of vitriolic ammoniac.---This triple salt crystallizes in molecules so small, as to render it impossible to determine their figure. It has a pungent, austere, and metallic taste. It decrepitates and is decomposed in the fire, giving the following products: 1^o volatile alkali; 2^o phlogisticated air; 3^o running mercury; 4^o volatile alkali combined with sulphureous acid. What remains behind in the retort is turbith mineral.--The ammoniated vitriol of quicksilver is but little soluble in water; this solution is precipitated white by alkalis and lime. This salt is completely decomposed by the marine acid, with which it yields corrosive sublimate, sal ammoniac, and vitriolic ammoniac.

moniac. By this analysis it was found that 100 parts of ammoniated vitriol of quicksilver, precipitated by water from its ammoniacal solution, contain 18 parts of vitriolic acid, 33 of volatile alkali, 39 of calx of mercury, and nearly 10 parts of water.

ERRATUM in the last Number.

Page 111, line 15, for "*Volatile sulphureous vitriolic acid*," read "*sulphureous or volatile vitriolic acid*."

XVIII. L I S T
OF
BOOKS ON CHEMISTRY,

Published in London, since our former List*.

PHYSICAL and Chemical Essays (translated from the original Latin) by *Sir T. Bergman*. To which are added Notes and Illustrations, by the Translator. Vol. the third, 8vo.

Elements of Chemistry, by *M. I. A. Chaptal*. Translated from the French, by *W. Nicholson*, 3 vols. 8vo.

Chemical Annals, translated from the French, of *Messrs. Morveau, Lavoisier, Monge, Bertholet, Fourcroy, &c.* Vol. 1. 8vo.

Baron Inigo Born's New Process of Amalgamation of Gold and Silver Ores. Translated from the German, by *R. E. Raspe*, 4to.

A Treatise on the Origin and component Parts of the Stone in the Urinary Bladder, by *W. Austin*, M. D. 8vo.

Description of a Portable Chest of Chemistry, by *I. F. A. Gottling*, professor of Chemistry at Jena. Translated from the German. 8vo.

* See the First Vol. of this Journal, page 105.

XIX. REMARKS

IN CONFIRMATION OF A THEORY
 ON THE
 NATURE OF PYROPHORUS;

By Mr. SCHEELE.

IN the first number of the Supplements to the Chemical Annals, Mr. Gottling has, in a truly chemical manner, taken great pains to overturn my theory respecting the inflammation of pyrophorus. In my Treatise on Air and Fire I have shewn, that, without alkali no pyrophorus can be prepared from alum; for I could not produce any pyrophorus from alum that was free from alkali. To what I have thus advanced, Mr. Gottling sets out with opposing the authority of Mr. Wieglieb, who did not obtain any pyrophorus from vitriolated

Vol. II. No. 3. Q tartar

tartar and charcoal. Mr. Gottling, too, repeated this experiment with as little success; and hence it plainly follows that I must have imposed upon my readers. But further. He prepared some alum totally free from alkali; treated it with charcoal-powder, and---obtained in this instance a good pyrophorus. Now that it might not be said that the charcoal supplied any alkali he has recourse to a very ingenious expedient; viz. instead of charcoal, he employs the residuum which remains after the preparation of vitriolic æther. The result was the same; he obtained pyrophorus—and now my theory falls to the ground. And what follows from hence? I must have conducted my experiments badly; I must have told untruths; for pyrophorus may be obtained from alum without any alkali. Mr. Wiegler certainly did not calcine the mixture of vitriolated tartar and charcoal long enough; otherwise the experiment would not have failed; for I have repeated it at least 30 times, and always with success.

I take

I take one spoonful of vitriolated tartar reduced to fine powder, and three spoonfuls of charcoal-dust, mix them well together, and put the whole into a matrafs or phial, which I place in a crucible, filling up the space all round between it and the crucible with sand, and calcine by means of a strong fire for between two and three hours: I then close the mouth of the phial with a stopper made of chalk, and let the whole grow cold. I fancy Mr. Gottling has only repeated Mr. Wiegles words; for it is impossible that this experiment should have failed in his hands, when even inexperienced chemists so frequently perform it with success. But what shall I say to Mr. Gottling's second experiment? He certainly never made the experiment himself; for I deny that he ever obtained pyrophorus from alum perfectly free from alkali. Though I was very certain that what I have said is the fact, yet I could not refrain from repeating the experiment once more (I am now sorry that I threw away so much time on this subject)

ject) I prepared some alum the same as Mr. Gottling's; I precipitated it's earth with alkali, lixiviated it with boiling water, dried it, and dissolved it again in diluted vitriolic acid, evaporated the solution to dryness, mixed it with charcoal-powder, and put one half thereof into a phial; the other half I moistened with lixivium tartari, put it likewise into a phial, placed both phials surrounded with sand, in a crucible, and proceeded to calcination in the usual manner. But what was the result of the experiment? The substance contained in the phial without alkali, was very far from being a pyrophorus; it did not even become hot: Whereas the other phial afforded a very fine pyrophorus. This is incontrovertibly the fact. And who does not see that Mr. Gottling has been deceived? (since he was not aware of the alkali that is necessary to the crystallization of alum) And consequently my theory of combustion, in spite of the many new hypotheses that have been started, still remains unshaken. It is founded
on

on the nature and property of vitriolic acid to attract in the fire, by means of the alkali, more phlogiston than is requisite for converting it into sulphur. If moisture is afterwards applied, this attraction of the vitriolic acid to the superfluous phlogiston is destroyed, in consequence of the greater affinity which the alkali has to the moisture. If pure air is at the same time applied, this air unites with the superfluous phlogiston which is let loose; hence the heat, and subsequent inflammation of the charcoal. For, I have clearly proved that, no pyrophorus will take fire in a perfectly dry air; it is absolutely necessary that moisture be at the same time applied.

XX. AN ACCOUNT
OF
SOME APPEARANCES ATTENDING
THE CONVERSION OF CAST INTO MALLEABLE IRON;

By THOMAS BEDDOES, M.D.*

AN alteration has lately been introduced into our manufactories of iron, in consequence of which the reverberatory has been substituted in the place of the finery furnace. The new process is capable of being indefinitely varied. I have lately been favoured with an opportunity of observing one of these variations with every advantage I could desire. As in this method the changes undergone by the metal during the first series of operations lie perfectly open to inspection, a short description of them may not perhaps be unworthy the notice of philosophical chemists. I did not content myself with a single examination; and, for

* Philosophical transactions part second, for 1791.

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the sake of greater accuracy, I took minutes of the phænomena, and of the time when they occurred. A very intelligent workman was at the same time directed to answer all my questions, so that I enjoyed the benefit of his experience also.

In somewhat more than half an hour after it was put in, the charge consisting of two hundred weight and a half of grey pig iron was nearly melted. The workman now began to stir the liquid mass; for this purpose he used sometimes an iron lever, and sometimes a kind of hoe; but he first turned the flame from off the metal, which is done by letting down a damper upon the chimney corresponding to that with which ordinary reverberatory furnaces are provided, and by raising the damper of a second chimney, which proceeds immediately from the fire-place, and carries off the flame, current of air, &c. without allowing it to pass into the body of the furnace.

In fifty minutes from the commencement of the operation, the metal had become in consequence of the constant stir-

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ring,

ring, loose and incoherent ; it appeared about as small as gravel ; it was now also stiff, and much cooled.

Fifty-five minutes from the same period ; flame turned on again. Workman keeps stirring and turning over the metal ; in three minutes it becomes soft and semi-fluid ; flame turned off ; the hottest part of the mass begins to heave and swell, emitting a deep blue lambent flame. The workman calls this appearance fermentation.

One hour and one minute. Blue flame breaking out over the whole mass ; heaving motion also general.

One hour thirteen minutes. Metal full as hot, or, as the workman and myself both judged, rather hotter than at the instant the flame was turned off, though it is now a quarter of an hour since.

One hour eighteen minutes. Where there is no heaving and no blue flame, the mass is sensibly cooler, and only of a dull red heat.

One hour and twenty minutes. Workman observes that the metal sticks less to his tool. Pig iron, he says, fastens upon it immediately,

mediately, and must be shaken off by striking the other end with an hammer; as it approaches more and more towards *nature* (malleable iron) it adheres less; and when the tools come clear up out of the mass, he judges it to be fermented enough.

One hour and twenty-three minutes. Little heaving or blue flame; metal stiffer, and of a dull red; flame turned on and soon off again.

One hour and twenty-six minutes. By constant stirring the metal is become as fine as sand. Workman remarks, that the flame which re-appears over the whole mass, looks more kindly. It is evidently of a lighter blue colour.

One hour and a half. Flame turned on and soon off again. Mass ferments strongly. Hissing noise heard; this noise was distinguishable in some degree ever since the blue flame and heaving motion became visible, but always faint till now.

One hour and forty minutes. Less blue flame.

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One hour and forty-eight minutes. Flame twice turned on and off in this interval. Metal now clots, stands wherever it is placed, without any tendency to flow, and no liquid pig-iron now remains in the basin of the furnace; the mass has been constantly stirred and turned over.

One hour and fifty minutes. A little finery cinder appears boiling up amid the mass. Workman attributes the increase of the hissing to this.

One hour and fifty-three minutes. Scarce any perceptible blue flame or heaving. All the metal is now gathered into lumps, which the workman beats and presses with an heavy headed tool. He brings them successively into the hottest part of the furnace, into which the flame has been admitted. He now stops the port-hole in the door at which he had introduced his tool, and applies a fierce flame for six or eight minutes; the metal is then cooled.

These appearances, at least the most interesting of them, seem to admit of an easy explanation.

explanation; and I offer the following observations as supplemental to those for which we are already indebted to the Swedish and French chemists on this important branch of metallurgy. I assume the following propositions as already proved by these philosophers. 1. That cast iron is iron imperfectly reduced, or in other words, that it contains a portion of the basis of vital air, the oxygene of Mr. Lavoisier. 2. That it contains a portion of plumbago, with which grey cast iron most abounds. 3. That plumbago consists of iron united to charcoal. 4. That fixed air, which I would rather call carbonic acid air, consists of oxygene and the constituent parts of charcoal.

The heaving or swelling motion, so conspicuous in the process, is doubtless owing to the discharge of an elastic fluid; and the lambent deep blue flame, breaking out in spots over the whole surface, shews, that this elastic fluid is an inflammable gas of the heavy kind. That no doubt might be
left

left upon the former of those circumstances, I directed the workman to take out, at two different periods, a quantity of the metal where it was working most strongly. Both proved, on examination, to be spongy, cellular and full of bladder holes.

The heavy inflammable air, I imagine is produced in this manner. The oxygene of the imperfectly reduced metal combines with the charcoal to form fixed air; at the same time another portion of charcoal is thrown into an elastic state, that is, into inflammable air, and burns on the surface with a very deep blue flame, on account of the admixture of fixed air. The heat which is so obviously generated in the mass at the beginning of the fermentation, I attribute to the combination of the oxygene and charcoal; a fact which, with several others as I have already remarked on another occasion*, shews, if not the falshood, at least

* Chemical opinions of a Philosopher of the last century.

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the imperfection of the modern doctrine on the subject of heat. The acidifying principle, it would appear, has some power of generating heat independent of its condensation. Here abundance of elastic matter is discharged; yet, notwithstanding the heat absorbed by its formation, and that which flows out of the metal in all directions, the whole mass becomes hotter. The oxygene cannot be supposed to have much specific or latent heat, because it undoubtedly exists in the iron in a very condensed state. Neither does the appearance of the mass allow me to ascribe this generation of heat to the burning of the inflammable air at the surface, as will also be immediately evident for another reason. The less deep blue colour of the flame at a subsequent period in the operation is probably owing to the absence of fixed air, or at least to its being produced more sparingly, the oxygene being now nearly consumed. It will not appear surprising, that the oxygene in this case should be consumed before the charcoal

charcoal if it be considered, 1. That grey iron contains a large portion of plumbago; and 2. that fixed air contains a much larger quantity of oxygene than of charcoal; near three times as much, according to our best experiments on its formation; so that I ascribe the subsequent fermentation accompanied with the lighter-coloured flame almost entirely to the conversion of the charcoal into an elastic fluid. A very experienced philosopher, I am well aware, has asserted, that water is necessary to this conversion; an opinion concerning the justness of which I have long entertained great doubts. Whenever I have distilled charcoal per se, I have found the first portions of gas to contain fixed air; an appearance owing, as I believe, to the decomposition of water absorbed from the atmosphere; but after continuing the process for some time, there has still been a production of inflammable air; but from this neither lime-water nor milk of lime would absorb any portion, though when fired with vital or common

common air, it would produce fixed air; and if moisture was added to the charcoal, inflammable and fixed air would be generated anew. Moreover it appears, from the experiments of Dr. Austin and some others, that charcoal consists of the hydrogen and azote of the French chemists. How far it may be difficult or impossible entirely to convert charcoal in its ordinary state into gas, is a point I wish to see more fully illustrated by future experiments. At present it seems obvious, that the circumstances of the operation I have described are particularly favourable to this conversion: for 1. not to mention the violence of the heat, we have this substance in a very attenuated state, so that, very probably, the expansive power of fire is very little, if at all, counteracted by the attraction of cohesion, which cannot be said in the case of the most minute mechanical division we can effect. 2. The attraction of the particles of the iron for one another will produce an effort to extrude the intermixed particles of charcoal, and thus

thus enable it more readily to assume the elastic form.

Now, during the continuance of the lighter-coloured blue flame, the mass, as I observed, shews no power of generating heat within itself; a circumstance which indicates that the heat produced in the former part of the operation does not depend on the burning of the gas at the surface; and I think inspection will satisfy any one that it is produced in the heart of the mass. It may indeed be objected, that the metal, now brought nearer to the state of malleable iron, may require a greater supply of heat to keep it at the same temperature. It is less fusible, as we are well assured. By referring back to the minutes, it will be observed, how very often it was necessary to turn the flame upon the mass during this second fermentation, in order to keep it in a state in which it could be worked.

The very copious production of elastic fluids during an hour, and often during a much longer space, for in this instance the

process was remarkably successful and short, does not seem favourable to a late ingenious hypothesis, according to which water is the embodying principle of all elastic fluids. I have never indeed considered this as very probable, and, after the observations I have related, I see no means of defending it. Will it be said, that the pig iron, as being in some sort a calx of iron, contains water?

In annealing crude iron, with or without charcoal, it is well known to increase in all its dimensions. I have seen bars originally straight bent like an S, when long exposed to heat in circumstances where they could not extend themselves end ways. I suppose this phenomenon may be owing to a very small beginning of this fermentative motion, which acts as an internal principle of expansion. Cast iron bars, not in contact with charcoal, would, according to this supposition, by long annealing, lose of their weight; or if the heat was too low for the elastic fluid to be discharged from their substance, they would probably blister

like steel; an appearance undoubtedly owing to the generation of air. Mr. Horne in his essay on iron, somewhere remarks, that on opening these blisters he has heard a whistling noise as of air rushing out, or rather rushing in.

During the whole of this process, frequent jets of white sparks, of a dazzling brightness, played from the surface of the metal. They would have afforded an extremely beautiful spectacle but for the inconvenience of looking on so hot a mass. They arose, no doubt, from the burning of small portions of iron.

The effect of so much stirring as I have noted down does not require to be explained.

The workman was clearly of opinion, that the fermentation of hard or white crude iron is less than of grey in this process; a fact which perfectly coincides with the preceding observations, since that species contains less plumbago, or in other words less matter fit to produce elastic fluids. In

order to prove the extrication of fixed air during the fermentation of the metal, I once thought of introducing lime-water in an iron vessel within the body of the furnace; but when I considered that the fire place was not divided by any partition from the body of the furnace, and that the whole building was full of burned air, I omitted the experiment from a persuasion that, even if the lime-water should become turbid, the fixed air might come from another source.

I was not unmindful of the sulphur which exists, as I have reason to believe, in every form of iron manufactured with coaks. I cannot, however, ascribe any of the effects I observed to its presence. There can be little doubt, that some portion was perpetually extricated with the inflammable air during the whole process; for on dissolving pieces of the stamped, or rather the rolled iron, in weak muriatic acid, silver held in the extricated air was tarnished as much and as soon as by air from specimens taken

out of the furnace at different times during the process. I could not but conclude, that the tarnishing matter came from the iron, when I found the air from a solution of zinc in the same acid, incapable of producing the colour upon silver. The appearance, the want of a martial astringent taste, and the dissolving action of caustic alkali, led me to conclude, that the colour in each experiment with iron was derived from sulphur.

XXI. NEW EXPERIMENTS
WITH
C H A R C O A L;
By Mr. LOWITZ.

(continued)

§. XXXI.

CAMPHOR and its odour are not in the least degree altered by charcoal; when this last, however, is added to a solution of unrefined camphor in spirit of wine, it deprives the same of its yellow colour.

§. XXXII.

If, to a saturated solution of camphor in highly rectified spirit of wine, charcoal be added in a quantity sufficient to let it settle well; the camphor will crystallize in the clear solution above the charcoal, nearly in the same manner as sal ammoniac, in the form

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of

of plumous crystals, which, according as the weather is warmer or colder, will alternately disappear and re-appear.

§. XXXIII.

A mixture of six ounces of cream of tartar and one pound of charcoal-powder was put into a matrafs, and boiled strongly for some hours with ten pounds of water. After the space of twenty-four hours, I found in the midst of the charcoal-powder, which had settled to the bottom, the whole of the cream of tartar crystallized in regular, large, flat, transparent crystals, which were three quarters of an inch long, and in respect to their figure quite different from the common crystals of this salt.

§. XXXIV.

It is well known that cream of tartar, in the state in which it is commonly met with, contains an admixture of tartarified calcareous earth. In the above-mentioned experiment (§. 33.) this calcareous salt was also crystallized among the charcoal-powder,

der, in the form of roundish transparent polygons, of a remarkably beautiful gold-colour.

§. XXXV.

Four ounces of a very brown oily salt of amber, after being well triturated with two pounds of fine charcoal-powder, were put into a large retort; the remaining empty space of which was afterwards filled up with coarse charcoal-powder, and the retort thus prepared was placed in a sand bath. After a very strong and long continued heat there was collected in the neck of the retort not quite a dram of a salt of amber that was as white as snow, and almost inodorous. Hence it appears that this acid; too, is decomposed by charcoal. With a smaller quantity of charcoal than was here employed, the purification, no doubt, would go on properly.

As the salt thus obtained was perfectly free from oily matter, I tried whether it might not be altered by smoking spirit of

R 4

nitre;

nitre; but notwithstanding all the pains I took, I was as little able as Mr. Hermbstadt to convert it into acid of sugar.

§. XXXVI.

Flowers of benzoin obtained by sublimation, are rendered uncommonly white and shining, when treated with charcoal-powder in the same manner as the salts of hartshorn and amber (§. 14. and 35) In this case, also, too great a quantity of charcoal may, in consequence of its decomposing power, prove injurious. Under this purifying treatment the oil of benzoin comes over as clear and colourless as water; by length of time, however, it becomes yellow again.

§. XXXVII.

Acid of apples is entirely destroyed or decomposed by digestion with charcoal, so that nothing remains but mere water, with a very little extractive matter of the nature of a neutral salt.

XXXVIII.

§. XXXVIII.

Though honey, boiled with charcoal, is thereby deprived of its peculiar smell and taste, and also of its colour and slimy parts; yet if it is farther evaporated, after the separation of the charcoal-powder, it again recovers its brown colour.

§. XXXIX.

By trituration with charcoal-powder bugs (*cimices*) were entirely deprived of their bad smell.

§. XL.

Spirits distilled from malt or other grain shew by the smell, as many other persons besides myself have observed, that their strength is much increased by purification with charcoal, without the help of distillation; infomuch that persons who were not informed of the manner in which the purification was effected, have taken such spirits for rectified spirit of wine. In order to determine whether this was a real, or only an
apparent

apparent increase of strength, I undertook the following experiments.

§. XLI.

(1) I purified four pounds of common ardent spirits with sixteen ounces of charcoal-powder, then weighed out exactly twelve ounces from the same, and by means of dry salt of tartar, I deprived this spirit as carefully as possible of its watery parts. Thus I obtained thirty-one drams twenty grains of a perfectly dephlegmated spirit of wine.

(2) In the same manner I dephlegmated twelve ounces of unrefined spirits, and obtained thirty-two drams ten grains of highly concentrated spirit of wine, i. e. fifty grains more.

(3) This loss from the ardent spirits which had been purified by means of charcoal was further confirmed by the difference in specific gravity between the two kinds of spirit; for the weight of the unpurified was to that of the purified as 1,0000 to 1,0084. Charcoal, therefore, operates with
a de-

a decomposing effect upon ardent spirits, and the above-mentioned apparent concentration (§. 40.) can be owing to nothing but the development of the spirituous from the empyreumatic and oily parts. This power which charcoal has of decomposing spirit of wine (a property with which I was before entirely unacquainted) is equally proved by the following varied experiments.

§. XLII.

(1) I distilled twelve ounces of common ardent spirits without charcoal-powder or any other addition till I was satisfied that I had abstracted all the spirituous parts. From this distilled spirit, which had a very bad smell, I obtained in the manner above described (§. 41. No. 1.) thirty-one drams forty-five grains of highly rectified or concentrated spirit of wine.

(2) Twelve ounces of the same ardent spirits were distilled over an equal weight of charcoal-powder, till the powder became dry; from this distilled liquor, which was quite

quite free from any bad smell, I obtained twenty-eight drams forty-five grains of rectified spirit of wine : i. e. three drams less. This striking loss is owing to the employment of three times a greater quantity of charcoal in this experiment.

§. XLIII.

In consequence of the preceding observations, perhaps some persons may at first be dissatisfied with my mode of purifying ardent spirits : However, if they will but take into consideration the following circumstances, they will no longer be discouraged from applying charcoal to this purpose.

(1) For the complete purification of ardent spirits a much smaller quantity of charcoal-powder is required, than that which was employed in the two preceding experiments (§. 41 and 42). Thus, one part of charcoal-powder to twelve parts of ardent spirits will be quite sufficient.

(2) If

(2) If the charcoal-powder is added to the spirits only in a quantity just sufficient for separating all the empyreumatic parts, it is obvious, that this charcoal-powder becomes saturated by immediately attracting those oily parts, and consequently that its decomposing effect upon a small portion of the ardent spirit itself is necessarily prevented; for the charcoal is not capable of attacking the spirit itself, unless it be added in a proportion too large to be completely saturated by the oily, empyreumatic parts.

But even if a very small quantity of the spirit, in consequence of not hitting upon the right proportion of charcoal, should be decomposed; still this loss when compared with the far greater advantage derived from the complete purification, is too trifling to be considered as any objection.

§. XLIV.

Concerning the purification of ardent spirits by means of charcoal without distillation, and the time which the charcoal-powder, added in different proportions, requires

quires before it completely settles, I have made the following observations.

I divided ten pounds (troy-weight) of ardent spirits into ten different vessels, to each a pound; and added charcoal-powder in the following increased proportions.

(1) Half a dram of charcoal-powder produced scarcely any alteration in the smell, and the spirits had not become quite clear even after the space of six months.

(2) One dram occasioned scarcely any perceptible diminution of the smell; and the spirit did not become clear till after the space of four months.

(3) With two drams the spirit became clear in two months.

(4) Four drams occasioned a very perceptible diminution of the smell; and the powder settled completely in the course of a month.

(5) One ounce entirely took off the bad smell; and the spirit became clear in a fortnight.

(6) With

(6) With an ounce and a half the spirit became clear in eight days.

(7) With two ounces, in six days.

(8) With three ounces, in five days.

(9) With four ounces, in twenty-four hours; and lastly,

(10) With five ounces, in two hours. The proportion of charcoal-powder could not be further increased, on account of the thickness which the mixture acquired.

§. XLV.

Hence it appears that the more charcoal is added at once to the ardent spirit, the sooner the clarification ensues. It is true that a proportionately greater quantity of the spirit is, in this case, imbibed or retained by the charcoal-powder; but, on the other hand, fresh spirits may afterwards be added, in the same quantities, to this used or impregnated charcoal, so much the oftener, viz. until the charcoal, in consequence of its saturation with phlogiston, ceases to produce any further effect.

It

It is somewhat remarkable that ardent spirits which have been completely purified by means of charcoal, give out a fine odour exactly resembling that of peaches.

§. XLVI.

In the Pocket-book for Chemists (1789, page 173) it is observed that vinegar is considerably weakened by distillation with charcoal-powder. On this subject I have made the following experiments.

(1) I distilled twelve ounces of common wine-vinegar, in a sand-bath with a gentle heat, but without the addition of charcoal, till all the liquor was abstracted from the retort. The liquor contained in the receiver was very empyreumatic, and weighed twelve ounces six grains. Its specific gravity was to that of water as 1,008, to 1,000, and its strength amounted to four degrees*.

* For an account of Mr. Lowitz's manner of ascertaining the strength of vinegar, the reader is referred to the first Vol. of this Journal, p. 250.

(2) Twelve ounces of the same kind of vinegar were in like manner abstracted to dryness over twelve ounces of charcoal-powder. The distilled liquor, which was very weak, weighed eleven ounces seven grains and a half. From the residuous charcoal-powder I afterwards obtained, in the open fire, a dram of highly concentrated and very pure *alcohol aceti*, which was quite free from empyreuma. I added this to the former product, and found the specific gravity not above 1,004, and the strength only two degrees.

(3) I distilled to dryness twelve ounces of vinegar with only one ounce of charcoal-powder. This is nearly the proportion in which I employ the charcoal (and always with the best effect) in distilling vinegar in the great. The product, which weighed eleven ounces six grains, was exactly the same in respect to specific gravity and strength as the vinegar which was distilled by itself (No. 1) viz. 1,008, (spec. grav.) and 4 degrees (strength).

§. XLVII.

Thus charcoal-powder operates with a decomposing effect upon vinegar also ; only, however, as is the case with ardent spirits, when added in too large a quantity. Accordingly what we said before (§. 43.) when treating of ardent spirits, viz. that no harm can result from charcoal when added in moderate proportions, will equally apply here.

§. XLVIII.

The so called acid of wood, which I obtained by distillation from lignum vitæ or guaiacum, was, like vinegar, very readily purified, by distillation with charcoal, from the great quantity of empyreumatic oil which adheres to it. It thereby acquires the taste and smell of distilled vinegar ; and after concentrating it to a very high degree by one of my new methods, I have brought this acid of wood, as it is termed, to crystallize, in the same manner as vinegar, and have thus obtained a true glacial vinegar.

§. XLIX.

§. XLIX.

I was very eager to repeat professor Lichtenstein's curious experiments (Chem. Annals 1786, Vol. ii, p. 217) relative to the solubility of charcoal. The result was similar to his.

(1) Into a retort containing eight ounces of smoking spirit of nitre, whose specific gravity was 1.476, and whose concentration amounted to thirty-four degrees, I put two ounces of charcoal-powder. A considerable heat was produced accompanied with effervescence or ebullition. The retort was placed in a sand-bath, and after a receiver was fixed to it, a gentle heat was applied. An elastic vapour of a fiery-red colour was extricated so copiously that nothing else could be perceived; whence it became necessary to stop the fire. When all was settled again, I continued the distillation (with a very strong heat at last) till the charcoal-powder was completely exsiccated. Thus I obtained three

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ounces

ounces six grains of smoking acid, of a very beautiful dark-green colour; its specific gravity was only 1,313, and its concentration only twenty-five degrees.

(2) I intended to add to the residuous charcoal-powder, after it was become cold, eight ounces more of fresh smoking spirit of nitre; but scarcely had I poured upon it half an ounce, before a quantity of ignited sparks suddenly appeared in the retort, which broke in pieces, whilst the charcoal itself took fire. In this case, however, there was no detonation like that which usually happens with nitrous salts.

(3) The experiment with eight ounces of smoking spirit of nitre and two ounces of charcoal-powder was again repeated in the same way as No. 1. When the distillation was over, the residuous charcoal-powder was somewhat shining in its upper part, but not much caked or agglutinated; however, it had settled pretty firmly at the bottom of the retort. In order to see whether it had suffered any alteration from this first operation,

operation, I subjected very small quantities of it to the following trials :

(a) When thrown upon burning charcoal it did not produce the slightest detonation, nor did it give out any smell.

(b) Applied to the tongue, it did not give out any saline taste.

(c) From a diluted brown lixivium of acid of tartar it extracted all the colour in the course of five minutes, without the assistance of heat ; whence it appears that it had not yet lost its dephlogisticating power.

(d) The affusion of smoking spirit of nitre upon it was followed by an extrication of heat, but not by inflammation.

(4) This charcoal-powder, which did not yet seem to have undergone the least alteration, I put back again into the retort ; I then mixed together the acid liquors (whose specific gravity was 1,325 and whose concentration amounted to twenty-nine degrees) obtained in the distillations No. 3, and No. 1, and poured them upon the charcoal. After this second distillation to dryness, the spe-

cific gravity of the obtained liquor was found to be 1,317, and its concentration twenty degrees.

(5) After the third distillation, the specific gravity amounted only to 1,147 and the concentration to only fourteen degrees. The charcoal-powder did not even yet shew the least signs of solubility.

(6) As the distilled liquor was now become very weak, I employed in the fourth distillation nine ounces of common nitrous acid, the specific gravity of which was 1,258, and its concentration equal to twenty-four degrees. At the end of the operation, I found the specific gravity of the distilled liquor to be 1,226. The residuous charcoal-powder was now become very strongly caked, and full of cracks.

(7) After the fifth distillation I did not find any difference in the specific gravity of the acid. A saline sublimate was contained in the neck of the retort.

(8) After the sixth distillation, the acid, whose specific gravity was still 1,222, appeared

peared of a yellow colour. The residuum had now a shining-black appearance, like pitch; on pouring upon it some of the distilled liquor, a reddish-brown solution was obtained.

(9) After the seventh distillation the acid became still yellower, and its specific gravity was now reduced to 1,218. The residuum was now for the most part soluble in water.

§. L.

Upon charcoal thus reduced to the form of an extract, in which state it possesses an extremely bitter and very astringent taste, I have hitherto made only the few following experiments.

(1) It is as soluble in highly rectified spirit of wine as it is in water.

(2) A sufficiently diluted solution of this substance in water is entirely deprived of its colour and bitter taste by fresh charcoal-powder. It is rather a curious fact, that, in the humid way, even charcoal itself

should thus be dephlogisticated by charcoal.

(3) If the watery solution of the extract of charcoal is thoroughly evaporated in a sand bath, it froths or swells up at the last, and becomes perfectly dry; but by exposure to the air it very soon attracts moisture again. It loses, however, this deliquescent property, and its humidity is gradually dried up, if it is left exposed to the air for some months.

§. LI.

Hitherto I had only purified brown saline substances by means of charcoal; but by the following experiments I found I could purify in like manner a naturally dark-brown resin.

Six ounces of soft resin of jalap, obtained in the usual manner from its solution in spirit of wine, and freed by ablution with water from its extractive parts, were dissolved in six pounds of highly rectified spirit of wine. To the dark-brown filtrated solution I added a pound of charcoal-powder,

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der, and after shaking up the mixture a few times, I set it by in the cold. After the charcoal-powder had settled, I decanted the liquor, which was now become of a lighter colour, into another vessel. That I might lose as little of the resin as possible, I diluted the charcoal-powder with two pounds more of highly rectified spirit of wine, poured it out upon a strainer, and squeezed out all the liquor from it, as well as I could, with my hands. To these solutions, which were mixed together, I added a pound more of fresh charcoal-powder, and proceeded in other respects as before. By a third repetition of this process, with the same quantity of fresh charcoal-powder, the solution was at length rendered perfectly clear and colourless, like the purest spirit of wine. The charcoal-powder was again lixiviated with highly rectified spirit of wine. The whole of the colourless liquor, which amounted to twelve pounds, after being very carefully filtered through three folds of bibulous paper, was, with the addition of a pound of water,

put

put into a retort, and the spirit of wine was afterwards abstracted by distillation.

The liquor which remained in the retort amounted to a pound and a half, and had a milky appearance. I poured it, while it was yet hot, into a matrafs the neck of which had been broken off, and evaporated it a little more. The resinous parts settled to the bottom, as colourless as the finest Venice turpentine. The supernatant yellowish liquor was poured off, and the soft resin was well rinsed out with pure water, by which means it acquired a beautiful milk-white appearance. By evaporation in a water-bath, I afterwards brought it to a proper degree of hardness. Thus I obtained three ounces of almost perfectly white, brittle, resin of jalap, which, notwithstanding the great quantity of charcoal that had been used, had lost nothing of its peculiar smell.

§. LII.

In order to see whether charcoal operates with a decomposing effect upon resin also, I
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treated a properly diluted solution of resin of jalap, with charcoal-powder in the cold, until it was totally deprived of its colour. I then weighed out exactly equal quantities both of this solution, and of another solution that had not been treated with charcoal, and evaporated them separately to dryness. The weight of the two exsiccated resins was exactly the same; there was only a very striking difference in the colour.

§. LIII.

I was now desirous of seeing whether I could also deprive colophonium, by the preceding method (§. 51.) of its reddish brown colour. But as I found by a previous small trial with a few grains, that a very unusual proportion of charcoal-powder would be required for this purpose; and as, moreover, an extraordinary quantity of spirit of wine would be necessary for the due dilution of the resinous solution, I was deterred from prosecuting the experiment farther.

§. LIV.

§. LIV.

Æther which is tainted with a sulphureous smell, is not deprived of this smell by charcoal-powder. On the other hand, water that has been impregnated as much as possible with such kind of æther, immediately loses this smell, when charcoal-powder is mixed with it, and there only remains behind the peculiar smell of æther. In like manner, the sulphureous water or phlegm, which comes over after the distillation of Hoffman's Anodyne Liquor, is completely and quickly deprived of its sulphureous smell by treatment with charcoal.

§. LV.

Upon the volatile, penetrating, and strong smelling substance which is extricated during the distillation of fuet and of olive-oil, charcoal-powder has not the least effect.

§. LVI.

Refins and native balsams, dissolved in spirit of wine, are deprived of their colour,
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but not of their peculiar smell, by charcoal-powder. The substances operated upon were benzoin, asafoetida, resin of jalap, mastic, amber, balsam of Peru, &c.

§. LVII.

The smell of distilled or essential oils is, in like manner, not at all affected by charcoal, whether the oils themselves are immediately dissolved in rectified spirit of wine, or whether the spirit of wine is distilled along with the substances which contain the oils. I made my experiments upon oil of lemon-peel, oil of orange-peel (bergamot) oil of lavender, oil of turpentine, oil of carraway-seeds, spirit of lavender, spirit of rosemary, &c.

§. LVIII.

On the other hand, empyreumatic oils, dissolved in a sufficient quantity of highly rectified spirit of wine, are entirely deprived of their colour and smell by charcoal. The oleum philosophorum, however, seems in this respect to afford an exception. I made
my

my experiments upon oil of hartshorn, oleum tartari foetidum, and oil of guaiacum.

§. LIX.

Distilled waters are rendered completely inodorous by treatment with charcoal-powder. If to any of these distilled waters only just so much charcoal powder be added as will suffice for destroying the smell, the water will always remain turbid; but when a larger quantity of charcoal-powder is added, the water becomes perfectly clear and transparent. This circumstance seems to be owing to the tenacious slimy particles, by means of which the essential oils are kept diffused and suspended in distilled waters; hence the water cannot become clear, till the charcoal has been added in a quantity sufficient for the separation of the slimy matter.

§. LX.

It may at first appear surprising that water only, but not highly rectified spirit of wine,

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wine, is thus deprived of the odour which is communicated by essential oils. But this may be easily accounted for. The complete purification of a distilled water by charcoal powder is a proof that the fragrant oily parts are entirely absorbed. This effect takes place the more readily, since the union of the oily parts with the water is very slight, and, as it were, merely mechanical: whereas in the case of the spirit of wine, the oily particles, in consequence of their strong affinity to the spirit, are thoroughly and intimately combined therewith, so as to afford an instance of true chemical solution. This is still further confirmed by the following experiments.

§. LXI.

If the attraction of the essential oils to the spirit of wine be weakened by the admixture of a proper quantity of water, so that the solution shall become milky, and a sufficient proportion of charcoal-powder be now added; the weakened spirit of wine
will

will be completely deprived of its oily particles, and will be rendered perfectly clear and inodorous.

§. LXII.

Charcoal-powder not only deprives distilled waters (§. 59) of their smell, but also water impregnated with any sort whatever of empyreumatic oil. I tried in this way oil of hartshorn, oil of guaiacum, &c.

§. LXIII.

A watery infusion of asafœtida prepared by digestion, and a cold infusion of Virginia snake root and valerian, were entirely deprived of the smell peculiar to these substances, by charcoal-powder.

§. LXIV.

Mixed with the empyreumatic oils themselves, charcoal powder produces no effect; for it is not possible to add to these oils such a prodigious quantity of charcoal-powder, as would be necessary for absorbing all the empyreumatic parts which in this case exist
in

in such an undivided, condensed, and concentrated state.

§. LXV.

In like manner, the essential oils themselves suffer no loss of smell from the admixture of charcoal-powder.

§. LXVI.

A watery solution of opium is immediately deprived of its peculiar narcotic smell, by the admixture of charcoal-powder with it while it is boiling. The solution afterwards runs through the filter easily and clear.

§. XLVII.

Both white and red wine are rendered as colourless as water by charcoal powder.

§. LXVIII.

Upon the common essence of orange-peel, I have not found charcoal-powder to produce the least change, either in colour or smell.

§. LXIX.

On the other hand, it deprives essence of wormwood not only of its smell, but also of its colour.

§. LXX.

Both the spirituous and watery tinctures of saffron are in like manner completely deprived of their smell and colour.

§. LXXI.

The spirituous and watery tinctures of red saunders, cochineal, and gum-lac are also rendered quite colourless. This effect takes place very quickly in the instance of the spirituous tincture of saunders. In general charcoal seems to operate with peculiar force upon red colours.

§. LXXII.

All the calcareous particles are completely separated from lime-water by charcoal-powder ; so that it becomes quite tasteless, and is not rendered in the least degree turbid by the addition of acid of sugar.

§. LXXIII.

§. LXXIII.

Water saturated with fixed air is very quickly and completely deprived of the same by charcoal-powder; so that lime water is no longer rendered turbid by it.

§. LXXIV.

If a little dry charcoal-powder be introduced into a bottle (in which no water has been left) filled with inflammable air, and the bottle, whilst the finger is kept upon the mouth of it, be shaken a little, a small augmentation of the air will be perceived. On applying the flame of a candle to the mouth of the bottle, the air within it will immediately take fire and produce an explosion; a proof that it must have received some pure air from the charcoal-powder; otherwise it would not be capable of exploding.

§. LXXV.

Water impregnated with hepatic air is completely freed from the same by means of charcoal-powder

§. LXXVI.

Onions, after they have been well bruised or mashed, are quickly and completely deprived of their strong smell by mixture with charcoal-powder. The same thing happens with garlick; but for the complete suppression or privation of its smell, garlick requires a much larger quantity of charcoal, than onions.

§. LXXVII.

If a little charcoal-powder is introduced into a bottle that has been filled with smoke, and the bottle is afterwards shaken, the smoke will be entirely absorbed, and the charcoal-powder will thereby lose its dephlogisticating power upon every other substance. Hence we see how necessary it is, that charcoal which is prepared before-hand for any of these experiments, be kept from the access of smoke, and of what is the constant attendant on smoke, phlogisticated air.

§. LXXVIII.

Besides the different kinds of charcoal which I formerly mentioned (Chem. An-
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nals for 1788, Vol. ii. p. 134) I have found that the coally residuum after the preparation of dulcified spirit of nitre, and also the coally matter which remains behind after the distillation of Hoffman's Anodyne Liquor (these residua being previouslyedulcorated and calcined in a covered crucible) are equally as proper for the dephlogisticating processes, as charcoal itself.

§. LXXIX.

Concerning the thoroughlyedulcorated coally matter which remains after the distillation of Hoffman's Liquor, it deserves to be mentioned as a circumstance which is somewhat remarkable, that I have employed it with success, before it has undergone calcination, to deprive a lixivium of acid of tartar of its yellow colour; it produces this effect, however, very slowly. Hence it appears that vitriolic acid is capable of reducing inflammable substances to a perfectly charred or coally state*.

* In the 4th No. of the Chemical Annals for 1791, Mr. Lowitz has pointed out the principal circumstances upon which, after an

XXII. EXPERIMENTS and OBSERVATIONS
ON THE
COMPONENT PARTS OF THE
STONE IN THE URINARY BLADDER;

AND
A CHEMICAL COMPARISON OF THE
STONE WITH URINE AND MUCUS;

By W. AUSTIN, M.D.

(Extracted from a larger Treatise on this Subject)

CONCEIVING that his new opinion concerning the origin of the calculus vesicæ (viz. that it is formed generally in very small part, and often in no degree whatever from the urine as secreted by the kidneys, but chiefly from mucus produced from the sides of the different cavities

experience of four years, he has found the success of the dephlogisticating processes with charcoal to depend. To a want of information in these particulars are to be ascribed the failure and disappointment complained of by many chemists who have repeated his experiments. The circumstances so necessary to the success of these operations are reduced to two general heads; viz.

I. The

through which the urine passes) would at once be determined by ascertaining whether mucus or urine abounded most with the

I. The preparation and quality of the charcoal.

II. The mode of employing the charcoal, according to the different nature of the substances to be operated upon. This comprehends whatever relates to

(a) Bringing the substances to be purified, to a state proper for being acted upon by the charcoal,

(b) Adding the charcoal in due proportion.

(c) Adding it at a proper time.

(d) The separation of the charcoal powder from the purified substances; and lastly,

(e) The further treatment of the liquid substances after the separation of the charcoal powder.

I. With regard to the preparation of the charcoal, it is absolutely necessary to deprive it of all its oily matter by making it red hot throughout its whole substance. Charcoal that has been badly burnt gives out its oily empyreumatic parts, and at the same time a brown colour, even to water, much more to saline leys, when boiled therewith. No wonder, then, that liquors which are treated with such imperfect charcoal, instead of being made colourless, should become even browner than they would be without such treatment.

In the space of nearly a year after he first discovered the purifying property of charcoal, Mr. Lowitz used to be guided, in the selection of what he wanted for the dephlogisticating processes, merely by outward appearance and lightness. But as in this uncertain mode of proceeding, some badly burnt charcoal was easily picked out along with the good, his experiments sometimes proved abortive. He afterwards took the precaution of making the charcoal red hot throughout; and since that time (viz. for the last three years) he has always accomplished his end.

sublimate of Scheele; Dr. Austin was led to examine the residue left after evaporating urine; the residue after evaporating blood;

(1) The charcoal must be made quite red hot in a furnace, and those pieces which cease to give out any smoke must be taken out with tongs, and be laid to cool upon *clean* bricks. Left the larger lumps should not have undergone a red heat throughout and there should still remain a little phlogistic or empyreumatic matter in the heart or middle of them, it will be proper to break them into smaller pieces before they are taken out of the fire; for a single lump of such imperfectly charred coal would counteract or destroy, if not altogether, at least in part, the dephlogisticating effect of the rest of the charcoal powder with which it should chance to be mixed.

(2) In the next place, great care must be taken that, during the cooling, the charcoal do not come in contact with any greasy, oily, or other inflammable body; for the smoke which would thus be produced, would be absorbed by the charcoal, and render it unfit for the dephlogisticating processes. For the same reason, the presence of much smoke (in whatever way produced) in the place where the charcoal is prepared, would be highly prejudicial.

(3) As soon as the charcoal is become cool, the ashes which adhere to it, must be carefully blown off with a pair of bellows; it must then be pounded and passed through a fine hair-sieve, and kept for use in clean earthen or glass vessels, closely stopped, in order to keep out the air.

(4) Much depends on the fineness of the charcoal powder; for the finer it is, the greater number of acting surfaces are presented to the substances to be operated upon, and consequently a smaller quantity of the powder suffices. By an attention to this circumstance, a good deal of trouble and labour may be saved.

(5) The goodness of charcoal-powder, thus prepared, may be easily and quickly ascertained by a small trial, in the following manner:

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the incrustation upon walls where people make water, and the incrustation also upon teeth. All these were exposed to a red heat

ner: Put an ounce of the most empyreumatic and worst kind of ardent spirits into a phial, and add thereto about a couple of drams of the powder; then shake the whole well together. If the charcoal-powder is of a proper quality, the ardent spirits will immediately lose their bad smell.

II. Respecting the mode of employing the charcoal, it is to be remarked that a given quantity of charcoal-powder attracts only a limited quantity of inflammable matter, after its saturation with which it becomes incapable of any further action.

Hence, when any substance is to be dephlogisticated, it is better to add too much than too little charcoal-powder; for in the first case the end will be accomplished, whilst in the latter, there will be only a partial dephlogistication, or perhaps none at all.

And yet too great an excess of charcoal must not be employed; for, not to mention that the process is thus rendered much more troublesome, the substances that are operated upon are thereby in danger of being decomposed (See p. 244 and 245 of the present Number of this Journal, where the objections that might be made, in consequence of this decomposing effect of charcoal, to Mr. L.'s method of purification, are obviated.)

(a) All solid bodies that are to be purified by charcoal (except such as admit of being treated in the dry way, by sublimation) must be previously brought into a fluid state by dissolution in their proper menstrua, from which the undissolved and grosser parts must be afterwards separated by filtration.

Nor is it enough that the substance to be purified be barely dissolved in its proper menstruum; it is further requisite that its solution be duly diluted either with water or spirit of wine, according to the nature of the dissolved substance.

In

for many hours, and their several products were carefully observed; but it was found that none of them yielded the sublimate of

In some cases where an empyreumatic oil, or slimy matter, adheres too strongly to the substance to be purified, it is necessary to boil the fluid along with the charcoal-powder even to dryness, and to re-dissolve it afterwards, and this for several successive times, till the purification is completed.

(b) To find out the proper quantity of charcoal-powder in operations in the great, add it little by little to the liquor during the boiling, until it shall be found, by repeatedly taking out small portions and filtrating and examining them, that the liquor is become sufficiently pure.

To as much acid of tartar as is obtainable from 30lbs. of crystals of tartar we are directed to add six ounces of charcoal-powder, beginning to add some of it as soon as the liquor is set to evaporate, and continuing the addition till the acid is brought almost to the point of crystallization. In this way ninety-six lbs. of acid of tartar, in the highest degree of purity and of a perfectly white appearance, were prepared from 300 lbs. of cream of tartar.

In the distillation of vinegar, Mr. Lowitz adds ten or twelve lbs. of charcoal-powder to 150 lbs of vinegar, and distills to dryness in the open fire. Here the receiver must be frequently changed towards the end of the distillation, since there generally comes over at the last when the charcoal-powder is employed in this proportion, about six or eight lbs. of brown empyreumatic vinegar; which, however, may be afterwards purified by a similar distillation with fresh charcoal-powder.

A terra foliata tartari, of a snowy whiteness, may be obtained by adding, during the boiling, from four to six ounces of charcoal powder to the product from about 5lbs. of salt of tartar, saturated with vinegar previously distilled with charcoal, in the manner before mentioned.

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Scheele in any sensible quantity. Not having just at that time sufficient mucus from the bladder of a calculous person to make

(c) Dry volatile salts, such as flowers of benzoin, salt of amber, salt of hartshorn, &c. should be triturated with an equal quantity of charcoal powder, put into a retort, and covered over, to about a hand's-breadth, with coarse charcoal powder. In the case of acid salts the proportion of charcoal-powder must not be too great; otherwise a considerable loss will be occasioned.

In the purification of ardent spirits the following is the safest way of proceeding. After the spirit is put into the still, the charcoal must be added little by little, till it is found, by frequently taking out small quantities and diluting them with a little hot water, that all the burnt or empyreumatic smell is gone off. Then, and not before, we are to proceed to distillation.

In the case of salts which by evaporation without charcoal powder constantly become brown and empyreumatic, the charcoal powder must remain mixed therewith till they are brought quite, or at least very near to the point of crystallization.

(d) The separation of the charcoal powder is easiest and soonest effected by means of a linen strainer. When no more of the liquor will run through of itself, the remainder must be squeezed out as well as possible, and, after it has been passed through a fresh filter and is become quite clear, it may be mixed with the former liquor.

That nothing may be lost, the charcoal powder which remains upon the filter may be lixivated with water; but there are very few cases in which this last weak lixivium can be added to the former liquors; it must be previously evaporated by itself, and undergo the other subsequent treatment.

A principal rule to be observed is, to strain the liquors that are boiled with charcoal as hot as possible; for they are then in the most fluid state, and consequently the separation of the fine particles of charcoal takes place more readily.

(e) After

the experiment upon, Dr. Austin dried the mucus expectorated by a consumptive person, and also some mucus from a person's bladder not affected with stone, and exposed

(e) After the separation of the charcoal powder, those lixivia that are liable to be affected by the fire, must be evaporated only with the gentlest heat.

In all experiments with charcoal, in which any comparison is made, in respect to specific gravity, smell, taste, or colour, between the liquors before and after the operation, it is necessary to keep in mind what was added for their due dilution before the treatment with charcoal, and to make allowances accordingly.

If in the first attempt to deprive any substance of its colour by means of charcoal powder a person should not succeed, he must not immediately conclude that charcoal is incapable of producing such an effect upon that substance. The solution might not have been diluted sufficiently. Before any conclusion is drawn, it is necessary that the solution be diluted to the utmost pitch (so, however, as to let the colour be still discernible) and that the charcoal powder be added in the largest possible quantity. Should no alteration then take place, he may safely pronounce that the colour of the substance thus tried is not affected by charcoal.

Heat is not always required in experiments with charcoal. Cold admixture, assisted by agitation, should be first tried; if this should not succeed, recourse must be had to digestion and boiling.

There are cases in which, notwithstanding the presence of such parts as are actually attracted by charcoal, its employment would, in other respects, be improper. In these instances, therefore, before any operations in the great are attempted, a trial in small should be made before-hand.

Note by the Translator.

them

them to a red heat, but observed no sublimate to be produced from them.

Being then supplied with mucus from a man who frequently voided small stones, he separated the mucus from the filtered urine, with an intention of examining each of these apart; but previously to the examination, he tried to obtain the sublimate from the calculi of this man, and was surprised to discover, that these calculi, though urged with a strong heat long continued, gave no trace of the sublimate. He immediately tried another calculus, which did not dissolve in caustic alkali, with the same event.

Hence the doctrine of the universal presence of this sublimate in urinary calculi, which has been generally adopted for some years, appears to be ill founded.

Inquiring into the difference of calculi which did, and which did not contain the sublimate of Scheele, the Doctor was led into some observations on the nature of the sublimate itself, and into some distinctions of calculi, which seemed to deserve attention.

tion. The appearances in all the experiments were noted down as they occurred, partly by Mr. Abernethy, and partly by the Doctor himself.

Three hundred and twenty grains of powdered calculus (being part of two calculi, both compact and of a light brown colour) were introduced into an earthen retort. Soon after the application of heat, a light brown liquor arose, which tinged the vegetable blues green. Shortly after, crystals of volatile alkali were formed on the extremity of the neck of the retort. A smell resembling the prussic acid was now perceived; and a piece of paper, that had been dipped in alkaline liquor, being put into the neck of the retort, and being afterwards immersed in a solution of iron, a fine blue precipitate was produced. After the prussic acid had been formed for a considerable time, there was observed a dark brown liquor, which, with the prussic acid, continued to come over until the end of the operation. These were examined repeatedly, with the same appearances; and a strong

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Strong heat was kept up till nothing more was produced. After the operation, 90 grains of sublimate, and four grains of distilled liquor were collected; and there remained in the retort 40 grains of charry matter in fine powder.

In another experiment, the residue from 320 grains was 13 grains. In this experiment the aëriform products were, first fixed air; then inflammable air mixed with fixed air; and when there was a red heat, volatile alkali.

In order to ascertain whether the colouring matter of Prussian blue existed in the stone or in the sublimate before the application of heat, he examined each of these by mixing them with the alkali and green vitriol, but could not produce Prussian blue from either of them.

To determine whether the residue of urine resembled the calculus in this respect, 380 grains of the incrustation upon walls where people make water, were introduced into an earthen retort. The incrustation was so moist

moist as to stick in places to the neck of the retort. There came over a liquor of a light brown colour, which became darker, and then lighter, impregnated with alkali, which did not appear to be combined with prussic acid, as was observed in subliming the stone. Paper, moistened with solutions of green vitriol and caustic alkali, was repeatedly presented to it during the whole operation, but no Prussian blue could be formed. The fire was kindled at half an hour before eight o'clock, and at half an hour after one, neither air nor moisture came over, though the heat was very great. After the sublimation, the charry matter at the bottom of the retort weighed 78 grains. The helm of the retort was thinly incrustated with a black substance, of which only half a grain could be collected.

Thus both the products and the residue from this kind of stone, and from the incrustation upon walls, differ very widely from each other.

A glass

A glass retort was charged with a considerable quantity of the residue after evaporating urine. Accidentally, the weight of it was not ascertained. It soon smoked in the fire, and gave over volatile alkali and fixed air, both in greater abundance than was observed in any other sublimation. The volatile alkali incruited the lower part of the neck of the retort. After the operation had continued an hour and a half, inflammable air came over with the fixed air, and also a very small quantity of prussic acid. Three quarters of an hour after, a sublimate had formed in the neck of the retort, to which a strong heat being applied, air came over very rapidly, which seemed to be a mixture of phlogisticated and inflammable airs. Paper moistened with a solution of green vitriol and of a fixed alkali, was instantly turned black upon being presented to these fumes; it then effervesced strongly with acid, the black disappeared, and it exhibited a very slight blue tint. The residue in the retort after the operation, was 220 grains. The sublimate, partly black and partly white,

weighed 3,5 grains; the white part was nearly 2,5 grains, and dissolved in nitrous acid.

Whether this small quantity of sublimate was the same with that which Scheele obtained from the calculus, Dr. Austin cannot positively say; nor does he, indeed, think it material. The quantity of this sublimate is so inconsiderable, and the appearance of Prussian acid so trifling, comparatively with what is always observed when the stone which contains the sublimate of Scheele is strongly heated, that he thinks it may be safely inferred that the residue from urine, including the mucus commonly contained in it, possesses the elements of this kind of stone (which he believes is the most common) in very small proportion.

At the time that Dr. Austin was going to examine whether the mucus of a calculous patient contained this sublimate in greater abundance than the urine, he was interrupted (as has been already hinted) by discovering, that the stone itself of this patient did not yield the sublimate in question. He
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had before observed that this stone and the dried mucus of the man from whom it was obtained, did not yield this sublimate. Hence it became a general question, whether other stones, not dissolving in alkalis, contained the sublimate of Scheele?

Accordingly, 20 grains of a calculus which had been almost, if not altogether, unaffected by lime-water and soap-lees even assisted by heat, were introduced into a small glass retort; and into another was introduced an equal quantity of calculus which had yielded the sublimate in a former experiment; and the two retorts were placed on a level with each other, in a covered cast iron stove, where a moderately strong heat was applied.

The appearances were as follow. From the stone which did not dissolve in alkalis, volatile alkali soon came over in abundance, and a watery liquor. No sublimate lined the neck of this retort. On introducing paper moistened with solutions of green vitriol and of alkali, little or no blue appear-

ed. At a second trial, the paper effervesced strongly with acids, but showed no Prussian blue. At seven successive trials the appearances were the same. No sublimate was seen in the retort in any part of this operation; the neck of the retort was just tinged a very little way with a thin film. The residue was 16 grains.

From the calculus which had yielded the sublimate in a former experiment, volatile alkali did not arise so soon. Brownish fumes soon filled the body of the retort; and a brown and white sublimate lined the neck to its very extremity, in great abundance. On introducing paper prepared as in the preceding experiments, much Prussian blue was discovered. At a second trial, a very great quantity of blue appeared, and no effervescence when the paper was dipped in acids. At four successive trials the appearances were the same. At a 6th, 7th, 8th, and 9th trial there was a slight effervescence upon the application of acids to the paper, and a very deep blue. The residue was not
very

very accurately collected ; it fell short of three grains. The sublimate was found in flakes in the neck of the retort.

The residue in one instance is about three grains, in the other sixteen. By this criterion alone the two kinds of stone may be readily distinguished, by exposing them to a red heat in open vessels and weighing the residues. It is probable, however, that they are not always so distinct ; but that the proportion of the sublimate varies in different calculi.

Mr. Lane observed, at least 20 years ago, that the volatility of calculi bore some proportion to their solubility in alkalis. The existence of the sublimate, to which both these properties are owing, had been discovered a few years before.

The heat in which Mr. Scheele produced the sublimate was nearly the same as was applied in the above experiments : For he made use of a glass retort in this operation ; and in all these experiments when glass vessels were used, as much heat was applied as they would bear without fusion ; indeed

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they were often actually melted. Dr. Austin presumes, therefore, that as far as his tests of the presence of the sublimate go, there is no proof whatever of its existence in one of these calculi. But the heat applied to those calculi which Mr. Lane examined, was still greater; they were sent to Mr. Alchorne, assay-master, and were kept in his furnace from the lighting of the fire till the heat was sufficient for cuppelling, which requires a strong red heat. It cannot be supposed that the sublimate, a substance so volatile and so easily decomposed, could in any state of combination resist this degree of heat.

Lime-water acts upon the calculi which contain the sublimate, in a very particular manner. During its first action, white spots appear upon the stone, and after a short time, fine clear crystals shoot like stars from different points of the stone. After standing a long time in the same liquor, these crystals become of an opake whitish colour, and break asunder. The Doctor has not always observed the appearance of
crystals

crystals even in the same kind of calculi; but he did not fully ascertain the cause of this variation. Dr. Whytt has taken notice of these crystals. Lime-water has the same effect upon the sublimate; and these crystals appear only when those calculi which possess the sublimate, are mixed with lime-water; Hence it is inferred, that either the entire sublimate or something contained in it, is a constituent part of these crystals, and consequently necessary to their formation. This analogy in the operation of lime-water upon the stone and the sublimate seems to shew, that the latter is not merely a creature of the fire, or a modification of certain parts of the stone produced by heat, and which did not exist before the decomposition of the stone, but that it is actually present in the stone in the same state.

As the sublimate dissolves in the caustic alkalis, so also calculi containing the sublimate dissolve readily, and almost wholly in the caustic alkalis; which, on the other hand, act but weakly upon those that do not

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As the sublimate dissolves in the caustic alkalis, so also calculi containing the sublimate dissolve readily, and almost wholly in the caustic alkalis; which, on the other hand, act but weakly upon those that do not

contain the sublimate. These last are generally whiter than the rest.

These experiments and observations are followed by an analysis of the sublimate itself, which when subjected, by itself, to a gentle heat, yielded chiefly volatile alkali; and when urged with a violent heat, little or no volatile alkali, but prussic acid, and phlogisticated air.* In this and another experiment of the same kind, besides a white sublimate, there was also found in the neck of the retort a dark brown crust.

Ten grains of the matter which had undergone three sublimations were put into a glass retort with two drams of nitrous acid, and a little distilled water. An effervescence, and extrication of air (partly fixed and partly phlogisticated) ensued. After placing the retort in hot sand, more air, and afterwards nitrous acid, came over. The sublimate dissolved slowly, but perfectly. On continuing the heat, more fixed and phlogisticated air was extricated. When the whole of the acid was evaporated, and the

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charge

charge was become dry, a strong heat was applied; during which, sublimate arose and volatile alkali came over. When the heat was still further increased prussic acid was discovered. Thus it appears, that even after solution in nitrous acid, the sublimate from the calculus retains its property of yielding caustic volatile alkali and prussic acid.

When mixed with vitriolic acid, the calculous sublimate turned black; and on the application of heat it yielded first fixed air and common air; then fixed air with 4-5ths or 5-6ths of phlogisticated air; and lastly, on continuing the heat, an air of which 9-10ths were fixed air. The sublimate was perfectly dissolved.

The brown residue after the third sublimation treated in like manner with vitriolic acid, afforded the same results; whence it appears that there is no essential difference between the residues and the sublimates themselves.

XXIII: PROPERTIES
OF THE BEST SORT OF
ZEALAND MADDER;

By MR. WATT.*

I.

THIS madder is of an orange-colour inclining to a brown, in a coarse and somewhat coherent powder: exposed to the air it attracts moisture, and thereby loses its properties, so as to become unfit for the purpose of dying.

II.

It gives with water a brownish orange-coloured infusion; a large proportion of water

* These observations were first published in the 4th vol. of the *Annales de Chimie*, and have since been republished by Mr. Berthollet in his *Elemens de l'Art de la Teinture*. Tom. ii. p. 120.

The Translator's acknowledgments are due to Mr. Watt for his obliging communication of the additional remarks contained in the last section of this memoir.

is required to extract its colouring matter. Marggraf directs three quarts of water to two ounces of madder. Water extracts its colouring particles either with or without the application of heat; but it seems to give out a finer colour without heat; its decoction is browner.

III.

When the infusion or decoction are slowly evaporated in an open vessel, there forms upon the surface a film, which, after some time, sinks to the bottom of the vessel, and is replaced by fresh films that continue to be formed one after the other, till the evaporation is completed.

IV.

The extract thus obtained is of a dark brown colour; only a part of it is soluble in water, to which it communicates a brownish tint.

V.

If the infusion is set to digest for some days in a matrafs (or vessel of such a form
that

that the liquor which is converted into vapour may fall down again) and the mouth of the vessel is left open, it deposits deep brown coloured pellicles; the liquor remains with a slight tinge of brown, and the pellicles are difficultly soluble in water.

VI.

Alum gives with the infusion (II.) a very deep red precipitate, which is composed of pellicles, and the supernatant liquor is of a brownish yellow colour.

VII.

The mild alkalis precipitate from this last liquor a blood-coloured lake (Marggraf's lake) the redness of which is more or less intense, according to the quantity of alum that was dissolved therein. In this way a very good blood-coloured lake may be obtained; but it cannot, by any method yet known, be made to have the brightness of carmine: It is transparent in oil; but in water it is opaque and dead.

VIII.

VIII.

If too much alkali is added, the precipitate is redissolved, and the liquor becomes red.

IX.

The lake precipitated by the mineral alkali is not of such a fine colour as that which is precipitated by the vegetable alkali.

X.

Calcareous earth precipitates a lake of a darker and browner colour than that precipitated by alkalis, especially if used in the form of lime water.

XI.

If a few drops of lixivium tartari are added to the water which is used for making the infusion (II.) this infusion becomes of a deep brownish red colour.

1°. Alum precipitates from this infusion a dark brown-coloured lake.

2°. Acids

2°. Acids added in small quantity give it a yellowish cast; and when added in larger quantity, they give it a brownish yellow-colour, but without occasioning any precipitation.

3°. This infusion evaporated to dryness yields a gummy extract, which dissolves readily in water.

XII.

If the infusion (II.) is made in water slightly acidulated with any of the mineral acids, it is of a yellowish colour.

1°. By long digestion, this liquor becomes of a greenish brown, and its red colour seems to be destroyed.

2°. By the addition of an alkali its red colour is restored, and if the infusion is afterwards evaporated, it yields an extract which dissolves readily in water.

XIII.

If aerated magnesia is added to the water which is used for making the infusion (II.) this last is of a light blood colour; and when evaporated

porated it yields a deep red extract, which dissolves readily in water.

1°. If the solution of this extract is used as a red ink, and exposed to the light of the sun, it becomes yellow.

2°. Alum precipitates from this solution a small quantity of a bad-coloured lake.

3°. Alkalis give it a redder and more permanent colour.

XIV.

The infusion made with a solution of alum is of an orange colour.

This infusion, precipitated by an alkali, gives a lake similar to that of (VI.) but not of so good a colour.

XV.

A solution of sugar of lead added to the infusion (II.) forms a brownish red precipitate.

1°. A solution of quicksilver in dephlogisticated nitrous acid, gives a purplish brown precipitate.

2°. A so-

2°. A solution of green vitriol, a fine bright brown precipitate.

3°. A solution of white vitriol was not tried.

4°. A solution of vitriol of manganese gave a purplish brown precipitate.

5°. A solution of tin in aqua regia was not tried.

6°. Vitriol of tin was not tried.

7°. The preparation used by callico-printers gave a fine brownish-red precipitate.

XVI.

The infusion (II.) mixed quite hot with an infusion of cochineal, gave a brownish red precipitate inclining to a deep purple, which did not dissolve readily in water: on continuing the digestion, more of this precipitate was thrown down.

1°. A bit of cloth that had been soaked in the preparation used by callico-printers, being dipped in this mixture, took a brownish red colour; and after being boiled in a solution of soap, its colour became a pretty good crimson.

2°. The

2°. The solution of soap in which it was boiled became very red; it gave, however, to paper but a very indifferent colour.

XVII.

1. Cotton cloth soaked in the printer's preparation, dried thoroughly and boiled in decoction of madder, became unequally red, as did the whole liquor. The cloth was not firmly dyed, but lost its colour by the air and washing.

2. Cotton cloth prepared as above and dried, steeped in a warm mixture of ox-gall and water, washed out and dyed in a decoction of madder, took a good red colour, which stood washing and boiling with a weak solution of soap.

3. On pouring some of the printer's preparation into the mixture of ox-gall and water, a whitish and copious precipitate was formed. The remaining liquor was not examined.

Observation. The gall contained in cow dung and sheep dung, the infusions of which are used by the callico-printers, seems to se-

parate the earth of alum from its solution in the acetic acid, and to form a salt or matter difficult of solution in water, which serves as a basis to which the astringent particles of the madder adhere strongly, and probably form a triple combination with it.*

* Mr. Hauffman (Annales de Chimie for September, 1791) suspects that the root of madder contains a peculiar acid, which opposes the attraction and adhesion of its colouring particles to the stuffs that have been alumed, or otherwise prepared for being dyed with this root. Hence he accounts for the great advantage derived from adding calcareous earth to the madder-decoction; viz. he supposes that the chalk absorbs the acid of this vegetable, forming with it a compound that is insoluble in water: But he has not yet had leisure to make experiments in order to obtain this acid in a loose or uncombined state. In the mean time, this fact remains fully established, that, in dying with madder the colour is much improved by the addition of calcareous earth.

Note by the Translator.

XXIV. EXPERIMENTS

ON

M O L Y B D Æ N A,

WITH A

VIEW TO ITS REDUCTION;

By MR. HIELM*.

§. I.

AT the request of the late Mr. Scheele, and Sir T. Bergman (the former of whom sent him a quantity of the acid to be

* On this subject Mr. Hielm's experiments are so numerous, that if we were to relate them at full length, we should, for some time to come, leave no room in our Journal for other dissertations. Accordingly, we can only offer an abstract, to be continued in some future numbers, from Mr. H.'s papers, of which not less than 7 (originally published in the Swedish Transactions) have already appeared in Dr. Crell's Annals.

X 2

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operated upon) Mr. Hielm undertook a course of experiments for the reduction of the acid or earth of molybdæna.

He previously melted together, for several times, vegetable alkali with bullock's blood; he then added an equal quantity of microcosmic salt and a little tartar, and black flux. After the acid had been mixed up with fat or oil and exposed to the fire till the fat was burnt away, these fluxes were added to it, and the mixture (sometimes with a little common salt scattered over it) was put into a covered crucible, and exposed for some hours to the heat of a wind-furnace.

The minute reguli (if they may be termed such) which Mr. Hielm obtained in this manner from the small quantity of acid of molybdæna that had been sent to him, gave occasion to Mr. Bergman's account of the effects of fire upon this acid-earth in his Treatise on the Blow-pipe. A trace of sulphur and iron was observable in these reguli

guli, and was supposed to proceed from the earth of molybdæna itself, since the fluxes employed were quite pure. Properly speaking, therefore, these reguli were only a sort of matt.

§. II.

Some experiments were afterwards made upon molybdæna itself. Exposed to the blast of a forge, both in open and covered crucibles, it suffered but little alteration. After half an hour's blowing, it appeared in the form of an agglutinated grey mass. It emitted a smell like that of liver of sulphur.—Equal quantities of molybdæna and iron, exposed to a strong heat, gave a matt, which when broken had a white metallic appearance, with a strong cast of red. This matt was pounded, mixed with some fresh iron, and exposed to the fire again; but the product turned out similar to the former.

§. III.

By roasting under a muffle, the molybdæna lost 25 per cent. in weight. By this process it is deprived of its sulphur.

(1) Of this roasted or desulphurated molybdæna 10 aſs were put into a crucible, the inside of which had been previously besmeared about half way up with linseed-oil, and afterwards dusted over with charcoal-powder; a small thin plate of copper, which weighed 24 aſs, and exactly fitted the crucible, was then introduced, and afterwards covered over with a little charcoal-powder. After exposure to the blast for half an hour, the copper was found melted round the bottom of the crucible, from which it readily separated. It was easily cut, filed, and hammered, was become of a brighter colour, and was increased eight and a half per cent. in weight. A portion of the earth of molybdæna was melted to the bottom of the crucible. The copper after undergoing fusion

fion with fluxes, was still somewhat malleable, though it cracked on the edges. Dissolved in aqua fortis, it left behind a white sediment, which (like molybdæna) tinged glass of borax green.

(2) Melted with crude or cast iron, without any other addition, it yielded a tolerably malleable mass, which, however, was covered with little knobs and streaks that were attracted by the magnet, and yielded to the file. The iron gained an increase of seven per cent. in weight, and approached to the nature of steel. By fusion with black flux, glass, and borax, a button was obtained, that was somewhat malleable, and internally of a white colour. Dissolved in vitriolic acid, and precipitated with alkali, the earth of molybdæna discovered itself by the blue colour that was produced.

(3) Tin, melted with it, had a pure white appearance, was rendered tolerably hard, and split and flew into pieces under the hammer; but even the smallest pieces were

not attracted by the magnet. The increase of weight was 14 per cent. which, however, by subsequent fusion with fluxes was reduced to only four and a half per cent.

(4) Lead melted with it, was somewhat malleable, and of a bluish white colour. Fused with black flux and tartar, it did not yield a true button, but had rather the appearance of calx or ashes of lead, invelliped in scorix of a beautiful green colour.

(5) Gold suffered no alteration by fusion with the earth of molybdæna.

(6) Silver, melted therewith, was constantly found to suffer a loss of weight; and the button thus obtained, appeared rough and jagged. This button, when melted before the blow-pipe, discovered its alloy by its spirting, and by the variety of its colours.

§. IV.

All attempts to reduce the earth of molybdæna, with no other addition but charcoal-

coal-powder (in the manner adopted for the reduction of manganese) turned out unsuccessful, whatever degree of heat was applied, or how long soever it was continued. By the help of a magnifying-glass, indeed, Mr. Hielm perceived some extremely minute, shining, metallic globules, which, even after fusion with borax, were still distinguishable in the dark green glass that was produced; but no compact and perfect button was obtained in any of these experiments.

§. V.

Although the peculiar metallic nature of molybdæna seemed to be sufficiently proved by these experiments (not to mention those of Pelletier, Sage, Ilseman, and Heyer); yet, Mr. Hielm still continued to devise methods for effecting the reduction of this refractory substance, as completely as that of any other metal.

§. VI.

§. VI.

A principal circumstance to be attended to is, that the earth of molybdæna be completely freed from sulphur and vitriolic acid. By Scheele's method with nitrous acid all the vitriolic acid cannot be separated. The roasting, above-described, is troublesome, not altogether certain or effectual, and is besides productive of loss. It was, therefore, found better, to make a large crucible, covered with another, red hot, and then to introduce the molybdæna, immediately pouring upon it some linseed-oil. As long as the oil continues to flame, the under crucible must be kept covered with the upper one; which, however, is to be afterwards taken off till the smoke and smell from the oil are at an end. This is to be repeated four, five, or six times; viz. until the cooled mass will readily triturate to a fine powder in a glass mortar. In this way a yellow powder, with a considerable admixture, however, of unaltered molybdæna, is obtained. This powder

powder is again introduced into the still red hot crucible, and treated as before. After it has been thus alternately burnt and triturated three or four times more, the earth is, for the most part, rendered pure. It is then put into a retort, moistened well with linseed oil, which is afterwards evaporated or expelled by placing the retort on burning coals (care being taken that the mixture do not boil over), and the heat is continued till the retort becomes red hot (in which case we are to guard against its melting): After it is become cool, fresh oil is to be added, and it must be made red hot again; and this treatment must be repeated four successive times. After breaking the retort, the mass or lump contained in it, is found to be of a dark blue, shining, metallic appearance, especially in the smooth, compact surface which was in contact with the bottom or sides of the retort; internally the same appearances are observed, on the application of a magnifying-glass. But the reduction is still uncertain; for linseed oil distilled by

by itself, leaves a similar appearance on the sides of the retort ; And if the above-mentioned mass is pounded and put under a muffle, a very pure calx of molybdæna is obtained, with the slightest roasting. The loss in this case seldom exceeds thirty per cent.

(To be continued in a future Number.)

XXV. CHEMICAL NEWS.

SOME beautiful experiments have been lately made at Prague on the combustion of diamonds (*Annales de Chimie* for November, 1791). To the diamond, which is to be burned, is fastened a bit of iron-wire, which is then made red hot, and plunged into a jar containing dephlogisticated air. The fire from the iron wire spreads to the diamond, which burns with the greatest vividness. Some diamonds, such for instance as those which come from Brazil, cannot be made to burn by this treatment. On account of the dearth of these substances, the experiments have not yet been carried to any great length; but Mr. Landriani hopes to prevail upon the Emperor to have them repeated, with a view to ascertain the quantity and quality of
of

of the residues, the alteration which the air in which they are burned undergoes, and the cause of the great difference in the qualities of diamonds.

A very simple method is practised in Bohemia (*ibid.*) for colouring the surface of deal, or other soft timber, of a fine black colour. It consists in rubbing the dry planks or boards with a piece of oak. The friction must be performed very briskly, so as to excite a momentary combustion. The surface of the deal is thus converted into a sort of charcoal, which adheres firmly to the wood, and which takes a pretty good polish.

Mr. Fabbroni of Florence (*ibid.*) has discovered a new solvent of the elastic resin (elastic gum). It is petroleum rectified several times. The elastic resin is completely dissolved by it, without the assistance of heat, and without suffering any alteration of its properties. Mr. F. has further discovered a species of earth, from which may be made bricks light enough to swim in water.

Pliny, Varro, and Vitruvius have made mention of such bricks; but the method of making them remained lost till now. The weight of these bricks is to that of the lightest wood, as 7 to 8. Mr. Giobert, who sends this account, is at present unable to give any information respecting the nature of the earth of which they are made.

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